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**DNA-TR-95-104**

## **Advanced Simulator Insulator Program Second Annual Report**

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**June 1996**

**Technical Report**

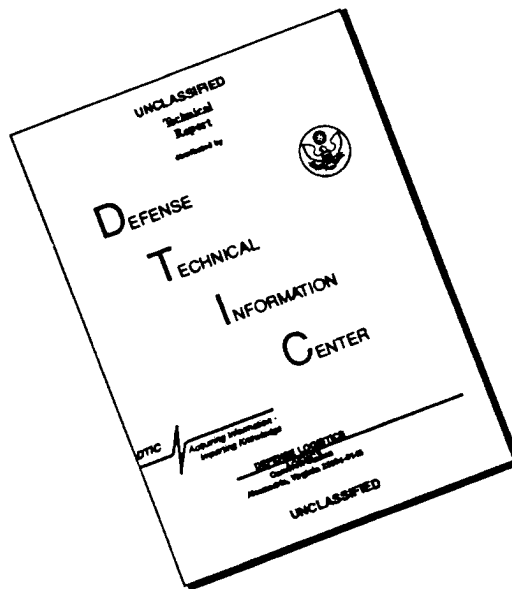
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# CONVERSION TABLE

Conversion factors for U.S. Customary to metric (SI) units of measurement.

MULTIPLY  BY  TO GET  
TO GET  BY  DIVIDE

angstrom	1.000 000 X E -10	meters (m)
atmosphere (normal)	1.013 25 X E +2	kilo pascal (kPa)
bar	1.000 000 X E +2	kilo pascal (kPa)
barn	1.000 000 X E -28	meter <sup>2</sup> (m <sup>2</sup> )
British thermal unit (thermochemical)	1.054 350 X E +3	joule (J)
calorie (thermochemical)	4.184 000	joule (J)
cal (thermochemical/cm <sup>2</sup> )	4.184 000 X E -2	mega joule/m <sup>2</sup> (MJ/m <sup>2</sup> )
curie	3.700 000 X E +1	* giga becquerel (GBq)
degree (angle)	1.745 329 X E -2	radian (rad)
degree Fahrenheit	$t_k = (t_f + 459.67)/1.8$	degree kelvin (K)
electron volt	1.602 19 X E -19	joule (J)
erg	1.000 000 X E -7	joule (J)
erg/second	1.000 000 X E -7	watt (W)
foot	3.048 000 X E -1	meter (m)
foot-pound-force	1.355 818	joule (J)
gallon (U.S. liquid)	3.785 412 X E -3	meter <sup>3</sup> (m <sup>3</sup> )
inch	2.540 000 X E -2	meter (m)
jerk	1.000 000 X E +9	joule (J)
joule/kilogram (J/kg) radiation dose absorbed	1.000 000	Gray (Gy)
kilotons	4.183	terajoules
kip (1000 lbf)	4.448 222 X E +3	newton (N)
kip/inch <sup>2</sup> (ksi)	6.894 757 X E +3	kilo pascal (kPa)
ktap	1.000 000 X E +2	newton-second/m <sup>2</sup> (N-s/m <sup>2</sup> )
micron	1.000 000 X E -6	meter (m)
mil	2.540 000 X E -5	meter (m)
mile (international)	1.609 344 X E +3	meter (m)
ounce	2.834 952 X E -2	kilogram (kg)
pound-force (lbs avoirdupois)	4.448 222	newton (N)
pound-force inch	1.129 848 X E -1	newton-meter (N*m)
pound-force/inch	1.751 2688 X E +2	newton/meter (N/m)
pound-force/foot <sup>2</sup>	4.788 026 X E -2	kilo pascal (kPa)
pound-force/inch <sup>2</sup> (psi)	6.894 757	kilo pascal (kPa)
pound-mass (lbm avoirdupois)	4.535 924 X E -1	kilogram (kg)
pound-mass-foot <sup>2</sup> (moment of inertia)	4.214 011 X E -2	kilogram-meter <sup>2</sup> (kg*m <sup>2</sup> )
pound-mass/foot <sup>3</sup>	1.601 846 X E +1	kilogram/meter <sup>3</sup> (kg/m <sup>3</sup> )
rad (radiation dose absorbed)	1.000 000 X E -2	** Gray (Gy)
roentgen	2.579 760 X E -4	coulomb/kilogram (C/kg)
shake	1.000 000 X E -8	second (s)
slug	1.459 390 X E +1	kilogram (kg)
torr (mm Hg, 0° C)	1.333 22 X E -1	kilo pascal (kPa)

\*The becquerel (Bq) is the SI unit of radioactivity; 1 Bq = 1 event/s.

\*\*The Gray (GY) is the SI unit of absorbed radiation.



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# SECTION 1

## INTRODUCTION

The major objective of this program is to improve the lifetime of vacuum insulators. We are doing this by developing a dendrite-resistant insulator coating that, unlike the Limitrak presently used, will not chip.

During the past year, the second of the program, Westinghouse formulated different coating materials and tested them for mechanical impact strength. The most promising were tested at Texas Tech for dc vacuum flashover strength. (We anticipate that dc vacuum flashover strength will scale to the pulsed times used in x-ray simulators.) The best two materials were then tested for dendrite resistance at MIT, and we selected the best one.

The best coating material tested has an impact resistance 80 times greater than Limitrak. Its dc vacuum surface flashover resistance is 27% greater than Limitrak, and it has greater dendrite resistance as well. We will test insulator rings coated with this material on PITHON.

In addition to developing an insulator coating, we are also developing envelopes for gas switches. During this year, Westinghouse made urethane envelopes for the DECADE Module 1 output switches. We will be testing these over the next year as well.

## SECTION 2

### FAILURE MODES FOR VACUUM INSULATORS

The present limit to insulator lifetime is chipping of the Limitrak coating. This is probably a mechanical failure. The insulator can be patched, but eventually it will break down at the patch sites. Mechanical failure is one of the four main causes of vacuum insulator failures. All four causes of failure are listed below, along with the means to avoid each failure.

- Mechanical fracture, caused by shock from arcs in the water upstream of the insulator. Avoid by using a sufficiently tough materials, such as urethane and new coating material which we have developed.
- Surface tracking during the main power pulse. In general, this does not cause damage to the insulator. However, it does limit the power to the load. Avoid by using an insulator stack long enough to limit the average electric field. The electric field must also be graded well enough so only a few rings flash during the main pulse. If too many rings flash, the electric field on the remaining ones will be substantially increased, causing the whole stack to break down.
- Dendrite damage caused by electrons. Avoid by using a dendrite-resistant coating, such as Limitrak or the new coating we have developed.
- "Worm-hole" discharges through the bulk of the material. This failure occurs in the following manner. In general, insulator rings flash on reversal of voltage if not during the main pulse. The first rings to break down flash over on the surface, since the surface breakdown strength is less than bulk breakdown strength. However, the last insulator will break down through its bulk if the electric field is large enough. This is because bulk breakdown occurs very quickly, while the surface flashover takes some time to develop. Avoid by using sufficiently thick insulator rings.

## SECTION 3

### INSULATOR DEVELOPMENT

#### 3.1 COATING DEVELOPMENT.

Different formulations of non-chipping, dendrite-resistant coatings were made and tested at Westinghouse. Westinghouse selected these formulations based on mechanical impact and 60 Hz arc resistance testing. The final compounds selected and their test results are described extensively in Appendix A. The results are summarized in Table 3-1, which lists the impact resistance along with the dc vacuum surface flashover strengths (discussed in the next section). Note that Compound 86 was ultimately selected as the best material.

Table 3-1. Average dc vacuum flashover strength and impact resistance for selected compounds.

Compound	$\langle V_{bd} \rangle$ (kV)	$\sigma$ (kV)	impact resistance (arb)
68, 1st test	23.6	8.5	216
86	23.3	4.8	360
68, 2nd test	22.7	6.3	216
69	21.8	6.2	432
72	20.5	5.8	264
urethane/alumina	19.0	5.8	—
Limitrak	18.3	4.4	4.5
urethane	16.8	—	—
85	16.3	2.7	132
73	15.5	4.5	312
82	15.5	2.5	264
88	15.0	3.9	360
89	13.1	3.9	360
71	12.1	4.2	432

#### 3.2 DC VACUUM SURFACE FLASHOVER TESTING.

Since all of the compounds selected have an impact resistance much larger than Limitrak, they were ranked based on their dc vacuum surface flashover resistance. (We are assuming that the performance ranking made with dc testing will scale to the pulsed discharges used on x-ray simulators.) The testing was done at Texas Tech; the process and the initial results are described in

Appendix C. Compounds 59 (identical to Compound 70) and 63 almost totally lost their flashover resistance after seven shots. However, the discharge occurred above the surface of the material, which suggested that the breakdown was due to outgassing.

To eliminate this, Westinghouse degassed samples by baking them under circulating air at 150°F for 18 hours. The samples were again tested, and this time did not show any sign of losing surface flashover resistance. As an example of this, the successive breakdown voltages for Compound 86 are graphed in Figure 3-1.

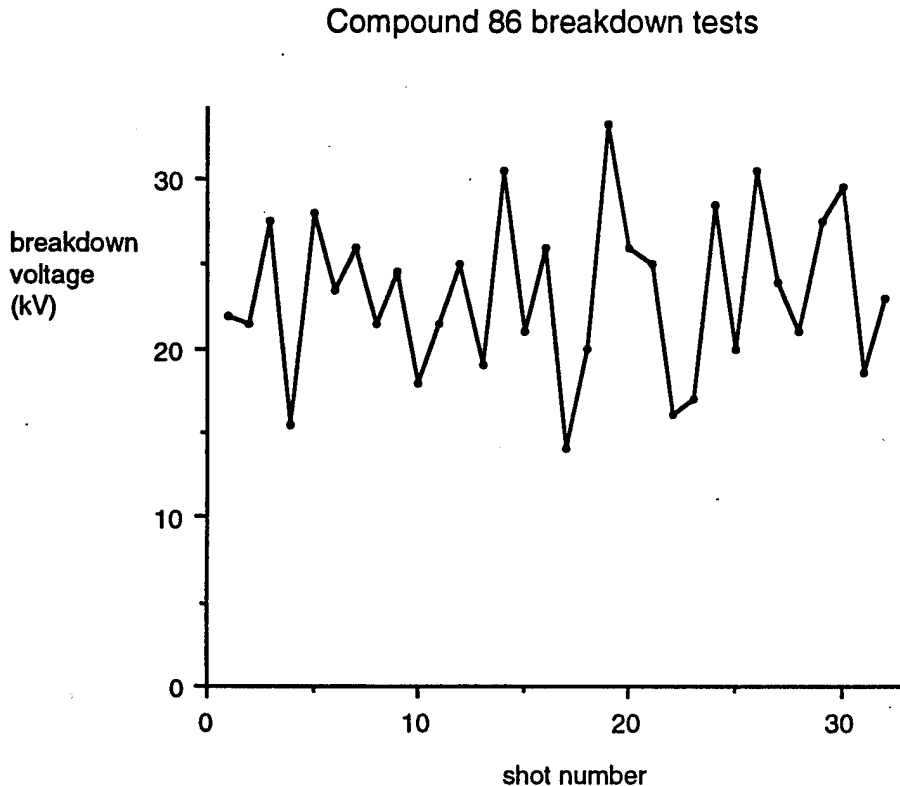


Figure 3-1. Successive measurements of the dc vacuum surface breakdown voltage of Compound 86.

Note that Compound 68 was tested twice. The first test series did not break down at the maximum charge voltage on the last three shots. This might have indicated an increased breakdown strength of this material over time. However, this was not the case; on the second test, all shots broke down, and the average breakdown voltage was less than for Compound 86.

### **3.3 DENDRITE TESTS AND MATERIAL SELECTION.**

We selected Compounds 68 and 86 as the two best materials, and tested them for dendrite resistance at MIT, along with Limitrak, Rexolite, and acrylic for comparison. The tests were done by irradiating the samples by an electron beam from a Vandegraaf generator. Because the formulations are opaque, any dendrites cannot be seen. Instead, the charge retained in the samples was measured; the greater the stored charge, the less the resistance to dendrites. The reason for this is that dendrites are formed by the sudden release of charge. The discharge creates a damage pattern which is similar to that of streams merging into a river.

Both Compounds 86 and 68 have dendrite resistance superior to Limitrak, which in turn is superior to Rexolite and acrylic. The dendrite testing method and the test results will be described more fully in the final report.

We selected Compound 86 as the coating to be tested on an x-ray simulator. Its dc vacuum surface breakdown resistance and dendrite resistance are as good or better than Compound 68, and it is markedly easier to machine. We expect that vacuum insulators using this new coating should have a significant increase in breakdown voltage compared to those using Limitrak or urethane.

## SECTION 4

### PITHON INSULATOR RING DESIGN

We chose PITHON as a testbed for a vacuum insulator coated with Compound 86. Two different insulator profiles have been used on PITHON previously. These are shown in Figure 4-1, along with the profile we will be testing.

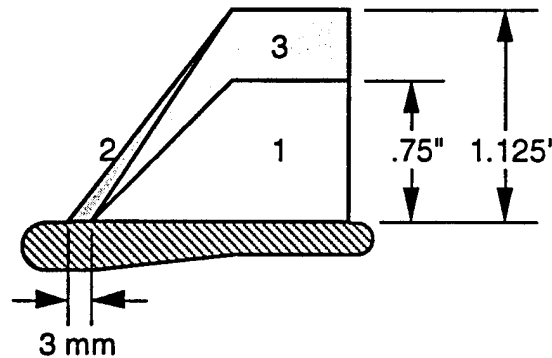
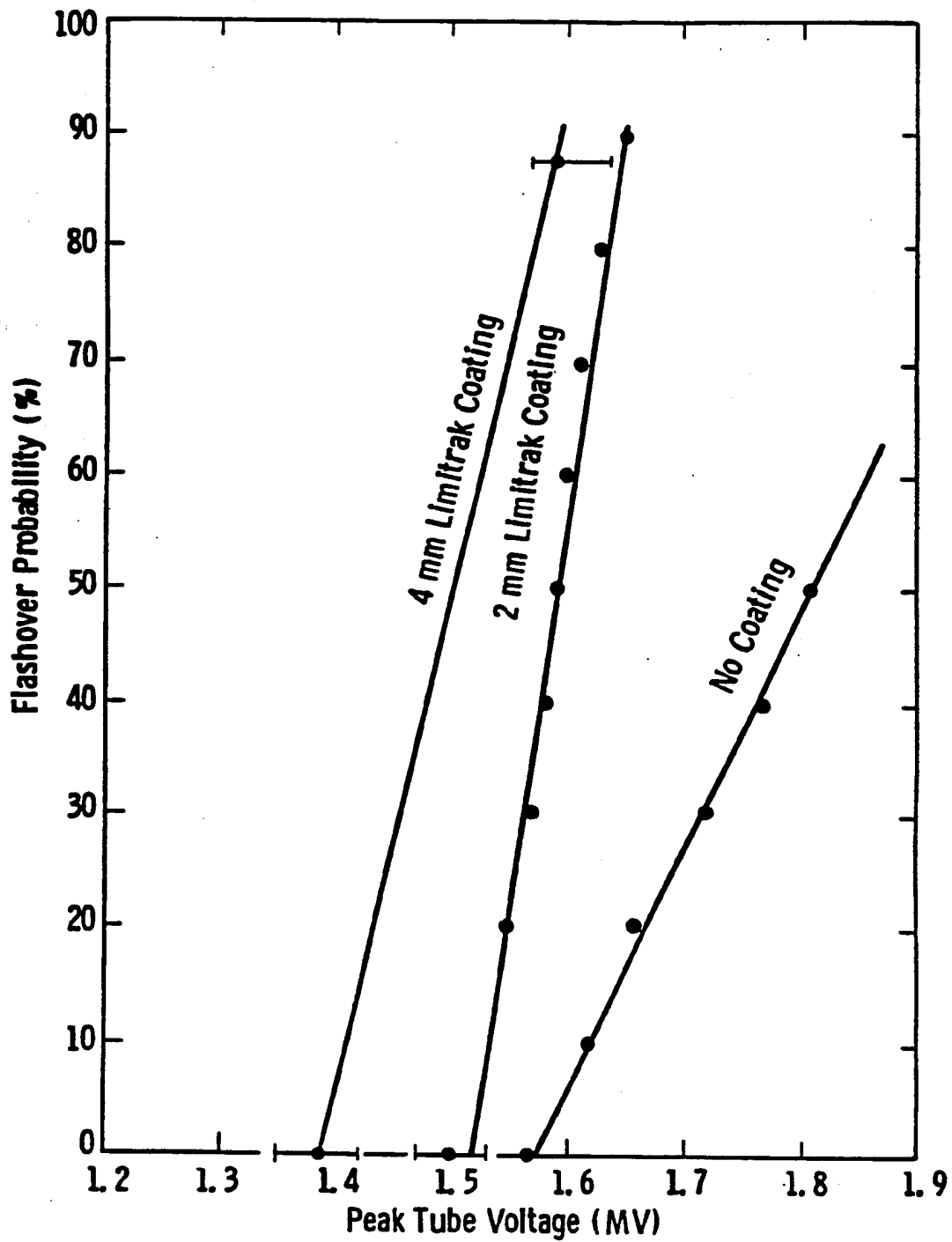


Figure 4-1. Insulator profiles used on PITHON. Profile 1 suffered worm-hole damage; Profile 2 did not, but had a low flashover resistance due to the inner extension. We will be testing coated insulators with Profile 3.

Urethane insulators with the first profile suffered worm-hole damage. To prevent this, taller urethane insulators with the second profile were used. These did not have any worm-holes. However, the vacuum insulator stack flashed at a lower voltage. This was because the insulators extended further inward by 3 mm. From the graph of Figure 4-2, we could have anticipated a 200 kV reduction in flashover voltage. The lower flashover voltage is probably due to increased electron bombardment of the insulator surface nearer the edge of the gradient ring. The insulators also had many dendrites; the damage may have also reduced the hold-off capability of the insulators.

To maximize the insulator lifetime and the flashover voltage capability, we will use coated urethane insulators which have the third profile. This profile has the same height as the second one, so it will not worm-hole. It also has the same inward extent as the first, to maximize the flashover strength.

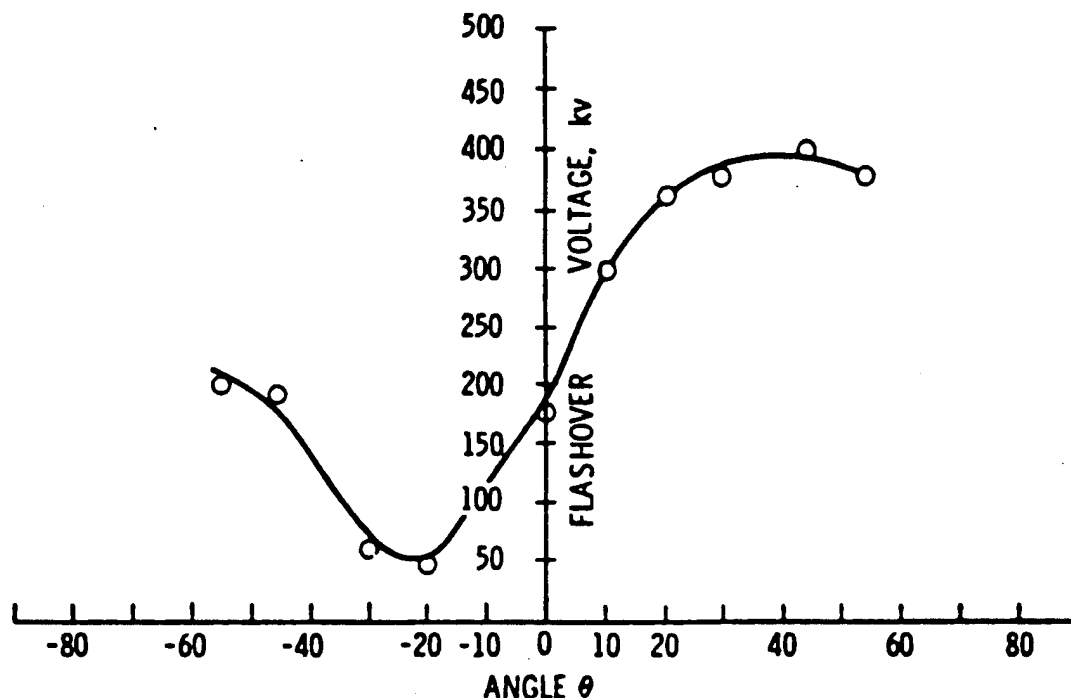




[J. Riordan, PI Internal Memo, May 2, 1988]

Figure 4-2. Probability of flashover of PITHON insulators plotted as a function of peak tube voltage. The two coatings extend inward by 2 and 4 mm.

Note that the third profile will have a slight decrease in flashover strength, compared to the ideal, because the insulator makes an angle with the vertical of  $39^\circ$ , less than the optimal  $45^\circ$ . Figure 4-3 shows the hold-off capability of an acrylic insulator as a function of angle. Assuming that the angular flashover dependence is the same for the coating as for acrylic, we can expect a 2.5% reduction in flashover strength, about 50 kV. We could reclaim this by making new gradient rings with a smaller inner diameter. However, we judged that the potential gain was too small to warrant doing this.



[O. Milton, Pulsed Flashover of Insulators in Vacuum, IEEE Transactions on Electrical Insulation, EI-7 (1), 9-15 (1972)]

Figure 4-3. Flashover voltage as a function of angle for acrylic. Pulse risetime about 50 ns.

At the end of this period, we began the process of making new vacuum insulator rings for PITHON. We machined the insulator ring blanks which will be coated, and began making test samples of the new coating material. We expect to be testing the insulator stack in early November of 1995.

## **SECTION 5**

### **URETHANE OUTPUT SWITCH ENVELOPES**

This program is also developing envelopes to be used for Marx and triggered gas switches. A problem with the acrylic envelopes currently used is that they can fracture. When this happens, the switch whose envelope has fractured gets soaked with the surrounding oil or water, and other switches in the system typically get wet as well. Cleaning the wet parts can take some time. To prevent the envelopes from fracturing, we can use a tougher material. Urethane and polycarbonate are two possibilities. At issue for these materials is the surface flashover strength. Westinghouse made six urethane envelopes for the DM1 output switches. They were able to eliminate bubbles by pouring the material into the bottom of the mold, as described in Appendix B. We will be testing these envelopes in the coming year.

## **SECTION 6**

### **CONCLUSIONS**

We developed a dendrite-resistant insulator coating that is superior to the Limitrak coating presently used because it does not chip. Its impact resistance is 80 times greater than Limitrak, its dc vacuum surface flashover resistance is 27% greater than Limitrak, and it has a greater dendrite resistance as well. We will be testing insulator rings coated with this material on PITHON during the next year.

We also made urethane envelopes for the DECADE output switches. We will be testing these next year as well.

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## **APPENDIX A**

# **IMPACT AND ELECTRON BOMBARDMENT RESISTANT PROTECTIVE COATINGS FOR PULSE POWER DIODE INSULATION - PART 3**

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Advanced Materials Technology

February 7, 1995



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## **ABSTRACT**

Candidate materials for use as an impact resistant replacement for the relatively brittle electron bombardment and arc resistant Limitrak have been developed; and samples from eleven of these compositions were sent to Physics International for evaluation. These materials were developed to function as protective coatings for the polyurethane insulator rings and discs in pulsed power generator diodes. Formulations, hardness, impact resistance and arc resistance of these alumina trihydrate containing materials are presented. Compared to Limitrak, these materials have up to almost two orders of magnitude greater impact resistance, and several had superior arc resistances.

Work has begun on developing a process for casting energy absorbing highly impact resistant decade module T.C. output switch envelopes from RBM 2-8-2 polyurethane. Three castings, which are in the form of hollow cylinders were sent to Physics International. Improvements in the process led to the preparation of an essentially bubble-free casting on the third attempt.



## 1. OVERVIEW

Eleven of eighteen candidate impact resistant epoxide based resin compositions cast in sheet form were sent to Physics International for evaluation of mechanical and electrical properties. These represented compositions having the highest impact resistance of those prepared thus far in this program. A sheet of cast Limitrak was also supplied for purposes of comparing properties. Compared to Limitrak, the candidate samples are considerably softer, have much higher elongations to failure, and lower tensile and compressive strengths. In addition to standardized static mechanical tests, we suggest that the glass transition temperature be measured, and dynamic mechanical testing (DMA) be performed in order to evaluate rate sensitivity phenomenon. Based on Physics International and Westinghouse STC test results, we will narrow the field to one to three candidate materials. We would like to subject these to some formulation optimization with regard to mechanical and electrical properties and processing. Processing studies would include both scale-up and reducing the outgassing of the materials. In order to conserve the resources for this program, we hope to perform this important outgassing investigation on a limited number of compositions.

Work has begun on developing a process for preparing a hollow cylindrical casting of RBM 2-8-2 polyurethane for use as an energy absorbing highly impact resistant decade module T.C. output switch envelope. The major problem area encountered thus far involves minimizing voids or bubbles in the casting. After initial attempts, an essentially bubble-free cylinder was made, which was sent to Physics International for evaluation. We expect to provide a detailed description of this effort in the next progress report.

## 2. EXPERIMENTAL AND DISCUSSION

### 2.1 MATERIAL PREPARATION AND DESCRIPTION

All of the epoxy specimens prepared during this report period contained aluminum oxide trihydrate (hydrated alumina) and were prepared according to the previously described procedure.<sup>1</sup> Their compositions are shown in Table 2.1.

A brief chemical description of the raw material components of the polymers has been presented in an earlier report.<sup>2</sup> During this period we have introduced another version of aluminum oxide trihydrate, Alcoa C-331. The principal difference between it and the previously employed Alcoa Hydral 710 is its seven-fold greater median particle diameter. A comparison of typical properties of these materials, provided by Alcoa's technical bulletins<sup>3,4</sup>, is shown below:

	<u>Hydral 710</u>	<u>C-331</u>
Typical Chemical Analysis (%)		
Al <sub>2</sub> O <sub>3</sub>	65.1	65.0
SiO <sub>2</sub>	0.001	0.001
Fe <sub>2</sub> O <sub>3</sub>	0.007	0.005
Na <sub>2</sub> O (Total)	0.30	0.14
Na <sub>2</sub> O (Soluble)	0.08	0.01
H <sub>2</sub> O	0.26	0.25
Surface Area, m <sup>2</sup> /g	5.5	3.6
% on 325 mesh	0.00-0.04	--
% thru 325 mesh	--	98
Median Microns (D50)	1	7

Table 2.1. Composition of cast sheets.

	67A	68A	69A	70A	71A	72A	73A	74A	75A	76A	77A	78A	79A	80A	81A	82A	83A	84A	85A	86A	87A	88A	89A
Epon 828	X	X	X																				
Epon 862				X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
Heloxyl 67	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
Jeffamine D-400	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
Jeffamine D-2000																							
Hycar 1300X16	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
Alcoa 710	X	X	X	X	X		X		X			X				X						X	X
Alcoa 331						X				X	X		X	X	X		X	X		X	X	X	X

The rationale for examining the larger particle size material is the expectation that it would provide a reduction in viscosity and thus easier processing. Preliminary work, although of insufficient quantity to draw firm conclusions, indicates that acceptable arc resistance (ASTM D-495) is obtained with the C-331 hydrated alumina. Concerning the effect of the particle size on mechanical properties, we would expect that finer particles would impart superior reinforcing. However, determining whether this effect is significant would require several replica and control samples because of the noise inherent in impact resistance tests. Such an evaluation could be performed when the formulation of the basic polymer has been fixed.

## **2.2 IMPACT RESISTANCE**

### **2.2.1 Crack and Cut-Through Measurements and Results**

Impact resistance tests were performed as described in a previous report<sup>2</sup> except that the 5.5 cm diameter disc specimens were cut from cast sheets having thicknesses ranging from 0.18 to 0.20 cm as opposed to the previously cast 0.51 cm discs. The impact resistance test data, along with hardness, are shown in Table 2.2

Specimens exhibiting superior results on our impact test were sent to Physics International for further evaluation of their mechanical properties. Selection was based on specimens achieving a figure of merit in excess of 200 as shown and defined in Table 2.2 The figures of merit values ranged from 216 to 432 as compared to Limitrak which displayed a value of about 4.5. Specimens were considered to have failed when they cracked or exhibited cut-through. Cut-through, as the term suggests, is considered to have occurred when the impact of the drop weight caused the circular edge of the sample retainer to cut-through the specimen. Although this is a different phenomena than cracking, it also provides an indicator of the ability of the material to withstand impacts from debris. However if "pure" shock is of dominant concern, then samples failing via the cut-through mechanism as opposed to crack formation may be preferred.

Table 2.2 — Hardness and Impact Resistance Tests

Specimen No.	Hardness (Shore A/D)	Drop Height (In.) Highest Passed, Failed	Drop Weight No.	Failure Mode	Figure of Merit
Limitrak	-/77	4,5	1	Cracked	4.5
67A	95/56	72,84	4	Cracked	312
68A	98/68	48,60	4	Cracked	216
69A	95/55	48,60	8	Cracked	432
70A	94/50	72,84	2	Cut-through	156
71A	94/55	48,60	8	Cut-through	432
72A	93/50	60,72	4	Cracked	264
73A	95/55	72,84	4	Cut-through	312
74A	94/52	24,36	4	Cracked	120
75A	95/60	60,72	4	Cut-through	264
76A	93/50	84,96	2	Cracked	180
77A	95/56	72,84	2	Cracked	156
78A	95/60	36,48	4	Cracked	168
79A	90/41	36,48	4	Cut-through	168
80A	92/44	84,96	2	Cut-through	180
81A	92/47	36,48	4	Cut-through	168
82A	94/55	60,72	4	Cut-through	264
83A	92/45	60,72	2	Cracked	132
84A	92/50	72,84	2	Cracked	156
85A	92/54	60,72	2	Cracked	132
86A	96/60	84,96	4	Cut-through	360
87A	96/60	84,96	2	Cut-through	180
88A	96/60	84,96	4	Cut-through	360
89A	98/68	84,96	4	Cut-through	360

Tests were performed on cast sheets  $0.19 \pm 0.01$  cm thick.

Figure of Merit = (No. of Wt.) (Avg. of highest passed and failed drop heights)  
for example for 89A, Figure of Merit =  $4(84+96)/2 = 360$ .

In the case of resisting impacts from debris, the situation becomes quite complex being dependent upon factors such as rate of impact, mass and physical properties of the impacting material, geometry of the collision, temperature, etc. Future tests should evaluate the effect on crack and cut-through susceptibility of the epoxy coating while being bonded to the polyurethane insulator ring. This bonding results when the epoxy layer is cast directly onto the polyurethane insulator ring.

### **2.2.2 Apparatus Errors**

As previously discussed<sup>12</sup> the impact resistance screening tests were developed to provide quick and inexpensive screening for a large number of experimental materials. Although test conditions were not precisely controlled, for example drop heights may have varied by about 0.5 cm (0.2 in.) when using the same guide tube, frictional losses which are believed to be relatively small were not determined for the various drop heights and weights, and typical daily variations in room temperature were of the order of one to two degrees Celsius, we nevertheless felt that the screening tests provided a useful indicator for differentiating the impact resistance provided by the various groups of formulations. In the case of the lightest weight, consisting of a sphere, we neglected possible rotational energy effects since the force applied by this weight was too low to cause cracking except for the brittle Limitrak. A comparison of damage resulting from dropping a weight twice as heavy from half the height was made for several materials and had displayed similar results as predicted by calculating the energy of the impacts with frictional effects being neglected.

### **2.2.3 Specimen Errors**

Stress concentrators caused by factors such as tiny bubbles or voids, specs from trace quantities of impurities and inhomogeneities even of minuscule proportions can reduce the impact strength of the specimen (certain highly dispersed inhomogeneities can, however, have the opposite effect). In addition,

stress caused as a consequence of polymerization and thermal shrinkage can render a specimen more susceptible to crack formation. The latter can often be reduced by employing optimum cure conditions, and as appropriate, by annealing the specimen. However, soft elastomeric materials can readily undergo stress relaxation and thus do not benefit from annealing as much as brittle polymers. Another factor leading to decreased reproducibility of impact resistance consists of surfaces which behave differently from the interior of the specimen. This can occur when surfaces of curing materials are exposed to the ambient hot air environment during cure. In such cases, surface evaporation of the more volatile components may occur before they react. Other surface effects can include reaction of amino groups with atmospheric carbon dioxide and of certain sites of the polymer with oxygen. Conversely, inactivation or loss of some of the curing agents may, due to upset stoichiometry, leave a soft layer on the surface which could absorb impact energy and thus give the specimen an improved impact resistance; however, such an effect would be difficult to reproduce in a reliable manner..

Care must be taken to insure that open dish castings of the same thickness are prepared on flat level surfaces. Some error results from meniscus and surface tension effects which cause a slight surface concavity. This effect becomes somewhat less important if the data are used for comparative purposes and are taken on the same type of materials which are prepared in the same manner. Sheet specimens cast between parallel vertical plates present less error.

Unfortunately, stress concentrators can have a very significant effect upon the accuracy of our test and any other type of impact test. Thus we feel that our test results should not be used to discriminate between small or even moderate differences but have merit when looking at major differences or "separating sheep from the goats". In our rough screening study, materials were tested singularly. Because of limited time and resources available, we chose to examine a wide variety of materials rather than fewer compositions with a statistically more significant number of replicas. We were also resistant to perform tests on a large number of

replicas of materials which, based upon subsequent testing of the sheet samples by Physics International, would not be of further interest. After narrowing the choice of candidates, we believe that perhaps 6 or more replica samples of each composition should be tested in order to increase the level of discrimination and testing reliability.

#### **2.2.4 Impact Resistance Phenomena**

Impact resistance, an obviously extremely important property for many applications, is a highly complex quantity which is difficult to define scientifically and its measurements often display poor reproducibility. There are a large number of impact tests which measure different quantities and depend on specimens of various specific sizes and shapes. Hence specimens are broken at different rates of impact and types of stress distributions.

Nielsen<sup>5</sup> in his Mechanical Properties of Polymers describes the well known Izod and Charpy pendulum type tests concluding that they "do not measure the true energy to fracture the specimen". In discussing falling weight tests he claims that "this type of test generally correlates much better with field tests and practical experience than does the pendulum type". However, just as there are many types of impact tests, there are many types of impacts that any given object may be exposed to in the field.

In falling weight impact tests, such as ours, besides the impact energy, which is proportional to weight and drop height, the velocity of the impact could be a concern for rate sensitive elastomeric materials. In our experience with rate sensitive materials at temperatures below and above the T<sub>g</sub>, an order of magnitude difference in rate does not usually have a very significant effect on the mechanical properties of the material. (Generally we looked at dynamic: static ratios of several orders of magnitude in studying rate effects in previous projects involving missile launch systems.) Since the velocity of a falling weight is proportional to the square



root of the drop height, we experience only a two-fold velocity increase in increasing the drop height from 6 in. to 24 in.

Besides the energy of the impacting mass, we are also concerned with the force of the impact. Since the force of the impact,  $F$ , according to Newton's second law, is given by the relation:

$$F = dp/dt$$

where the momentum vector,  $p$ , is defined as:

$$p = mv$$

The relations for energy and force in terms of drop height and weight are thus different. For example, unlike energy, the force resulting from dropping twice the weight from half the height will be greater. This was found to be the case in testing of rigid materials from another project. These materials failed at relatively low impact conditions purely by shattering and did not exhibit any evidence of even the slightest cut-through.

The derivative  $dp/dt$  represents a complicated phenomenon since it depends upon the nature of the collision. In addition to the drop conditions it is affected by factors such as the viscoelastic nature of the substrate which influences the velocity-time relation from the initial moment of impact. Further complicating the situation is that the partially elastic collisions exhibit, whether or not cracking occurs, varying degrees of cut-through.

### 2.3 ARC RESISTANCE

Arc resistance measurements were performed according to ASTM D-495 on retained specimens from the cast sheet samples sent to Physics International (Table 2.3). It is of interest to note that some of these candidates, besides being much more impact and chipping resistant than Limitrak, also displayed superior arc resistance.

Table 2.3 — Arc Resistance of Samples Sent to Physics International

Sample	$\text{Al}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$ , %	Density, g/cc	ASTM D-495, Seconds
Limitrak	48	1.67	150,140,134
67A	48	1.47	130,135,139
68A	48	1.39	142,143,146
69A	48	1.41	144,144,152
71A	48	1.38	140,140,141
72A	53.5	1.56	185,187,159,166
73A	53.5	1.53	178,183,175
75A	56.5	1.61	194,185,180
82A	56.5	1.58	183,183,183
86A	53.5	1.57	187,184,185
88A	48	1.43	140,142,140
89A	48	1.48	142,141,139

## 2.4 OUTGASSING

There is concern about outgassing of the samples sent to Physics International as evidenced by odor. Since our retained samples are stacked together, we could not determine whether the odor came from all or a few since the volatile odorous compounds would be absorbed by all of these materials. After spreading out the samples and baking them for 18 hours at 70°C in a vented forced circulating air oven, compositions 67A, 82A, 86A and 89A did not have a detectable odor and the odor level for the remaining had greatly diminished so that in the other cases we were not certain whether these samples were odorous. Upon examination of all the specimens after an additional two days in the laboratory, we could not detect any odor.

Besides baking the epoxy coatings under a stream of an inert gas or as described above, we would propose examining approaches listed below:

- (a) Vacuum distillation of the raw materials would remove volatile impurities, some of which may be nonreactive and thus could exhibit subsequent outgassing.
- (b) Altering the stoichiometry slightly could be helpful if the problem is incomplete reaction of all of the reactants.
- (c) Increasing the cure temperature and/or use of certain catalysts would also be helpful in solving problems related to unreacted reactants. However, care must be exerted in using catalysts so as not to decrease the working time of the mixture too severely. Overly increasing the cure temperature would likely result in a harder material with decreased impact resistance.

We suggest that Physics International bake the samples in a forced air circulating oven at 70°C for about 16 h and then proceed with evaluating them despite the odor; and on a narrowed field of perhaps 1 to 3 compositions we would proceed with the approaches suggested above.

## 2.5 FABRICATION OF POLYURETHANE SWITCH CASE

According to the overall objectives of this project, we are making blank RBM 2-8-2 polyurethane cylinders, that are to be machined as Decade Module Output T.C. switches. Presently, these switches which are made of polymethylmethacrylate (Plexiglas) have failed by mechanical rupture during switching. It is expected that RBM 2-8-2, with its superior mechanical impact properties will give switches with good operational properties that resist severe mechanical shock as was the case when it was used for rail gap switches.<sup>6</sup>

A mold for producing a hollow cylinder from which the impact resistant decade module T.C. output switch envelope can be machined has been fabricated. It consists of two concentric aluminum tubes recessed into a aluminum base plate and is shown in Figures 2-1 and 2-2.

We are working on developing the process so as to minimize bubbles or voids in the casting. Thus far three castings have been prepared. The first two were processed using a relatively simple procedure involving degassing of the freshly mixed prepolymer-extender mixture, pouring it into the mold and degassing again when the mold is about half filled and then filled almost to the top.

Because of concern with demolding, for the first casting we lined the mold with a thin Teflon film in addition to coating the metal with a release agent. Unfortunately, the Teflon film was not adequately secured in place and thus became embedded in the casting. For the second casting we depended solely on the mold release agent and were able to demold the cast cylinder. Unfortunately, in both cases the castings contained bubbles; however, otherwise the overall physical integrity of both castings appears good.

Efforts are underway to increase the degree of evacuation including the pre-evacuation step for the molten extender while it is at 140-150°C and the prepolymer while it is at its processing temperature of 100°C just prior to mixing. We have also designed our system to accommodate filling the mold from the bottom while under vacuum. These efforts resulted in our preparing an essentially bubble-free

casting, which along with the previous two has been sent to Physics International for evaluation.

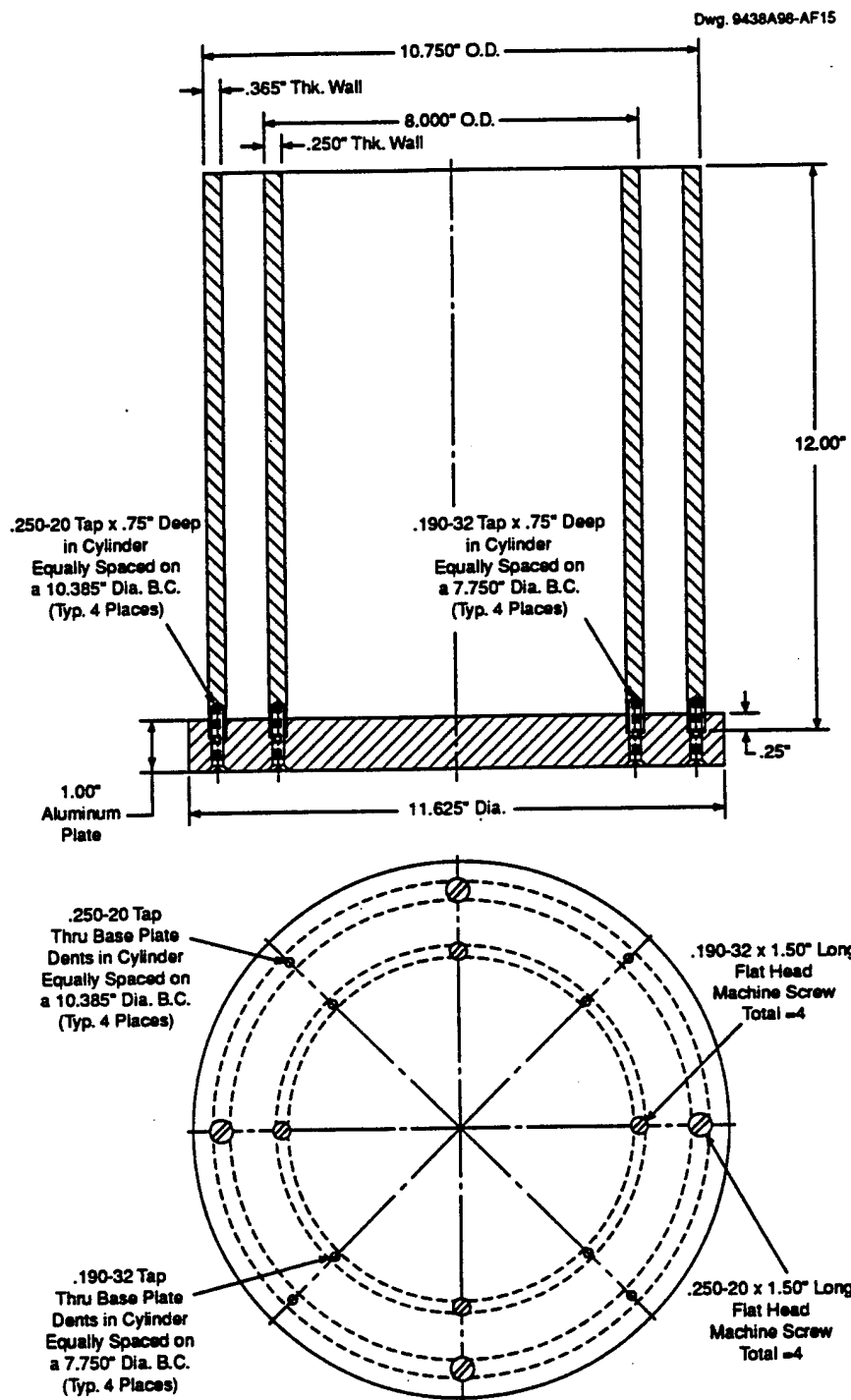


Figure 2.1 — Mold for Cast Polyurethane Decade Module T.C. Output Switch Envelope

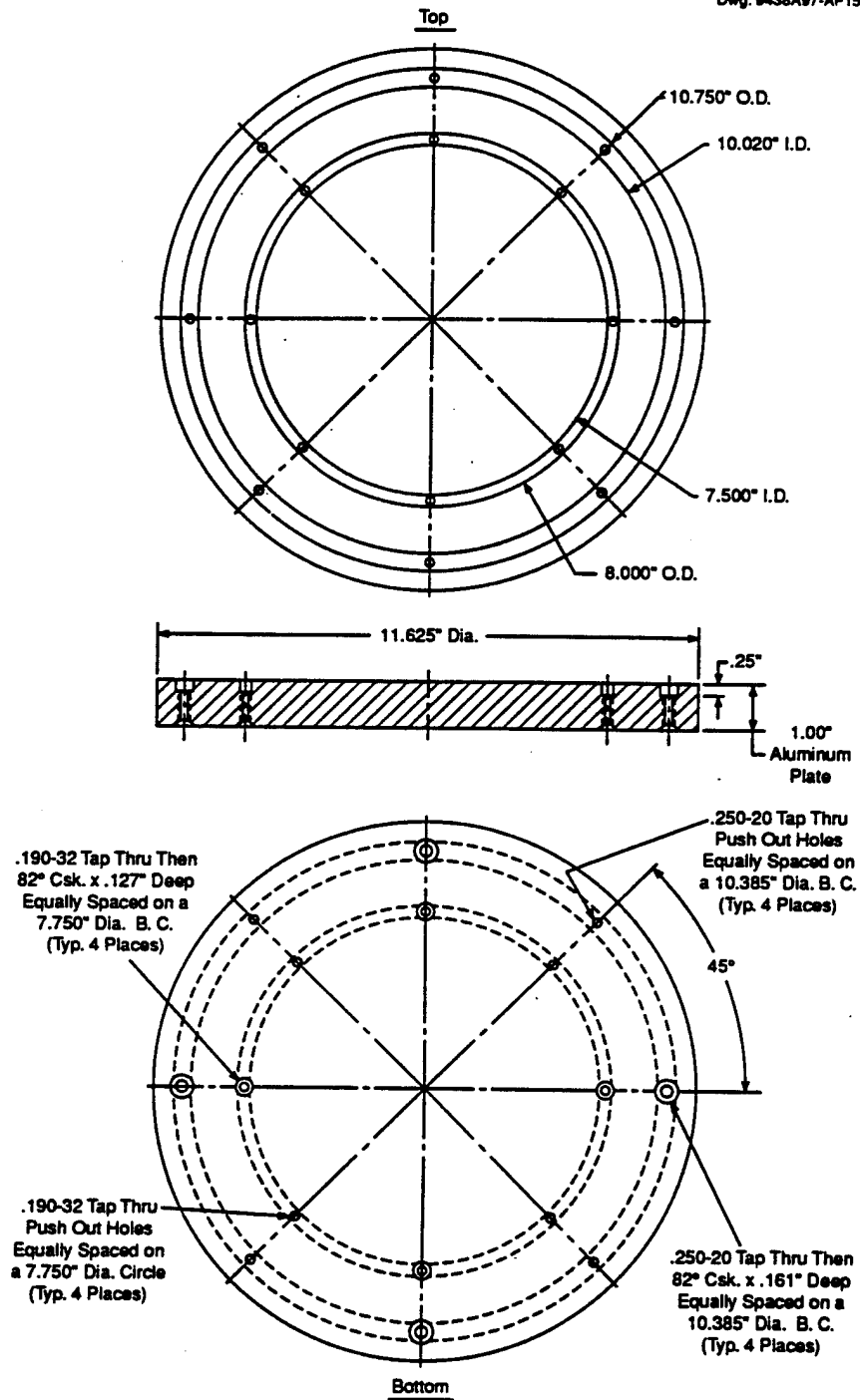


Figure 2.2 — Base Plate for Mold for Cast Polyurethane Module T.C. Output Switch Envelope

### **3. RECOMMENDED CONTINUED WORK**

- Reduce outgassing of epoxy coatings.
- Develop process for preparing a hollow cylindrical casting of RBM 2-8-2 polyurethane for machining into an energy absorbing, impact resistant decade module T.C. output switch envelope.
- Optimize chemical formulation of epoxy coating with respect to processability and mechanical and electrical properties.
- Examine use of a 50  $\mu$  diameter hydrated alumina powder alone and in combination with finer particle size material in order to obtain a superior combination of high filler loading and low viscosity.
- Prepare candidate epoxy coatings bonded to the polyurethane insulating resin for evaluation of impact resistance.



#### **4. ACKNOWLEDGMENT**

The authors wish to thank Leslie Kennedy for preparing the test specimens and performing the impact tests.

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## **APPENDIX B**

# **DEVELOPMENT OF A PROCESS FOR CASTING RBM 2-8-2 POLYURETHANE CYLINDERS FROM PULSE POWER DECADE MODULE T.C. OUTPUT SWITCH ENVELOPES**

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**Advanced Materials Technology**

**March 28, 1995**



**Westinghouse STC**

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## **ABSTRACT**

The development of the process employed in casting RBM 2-8-2 polyurethane cylinders for Pulse Power Decade Model T.C. Output Switch Envelopes is presented. The construction of the mold and the auxiliary equipment is described. Details are provided for synthesizing the prepolymer, preparing the extender, combining them and filling the mold.

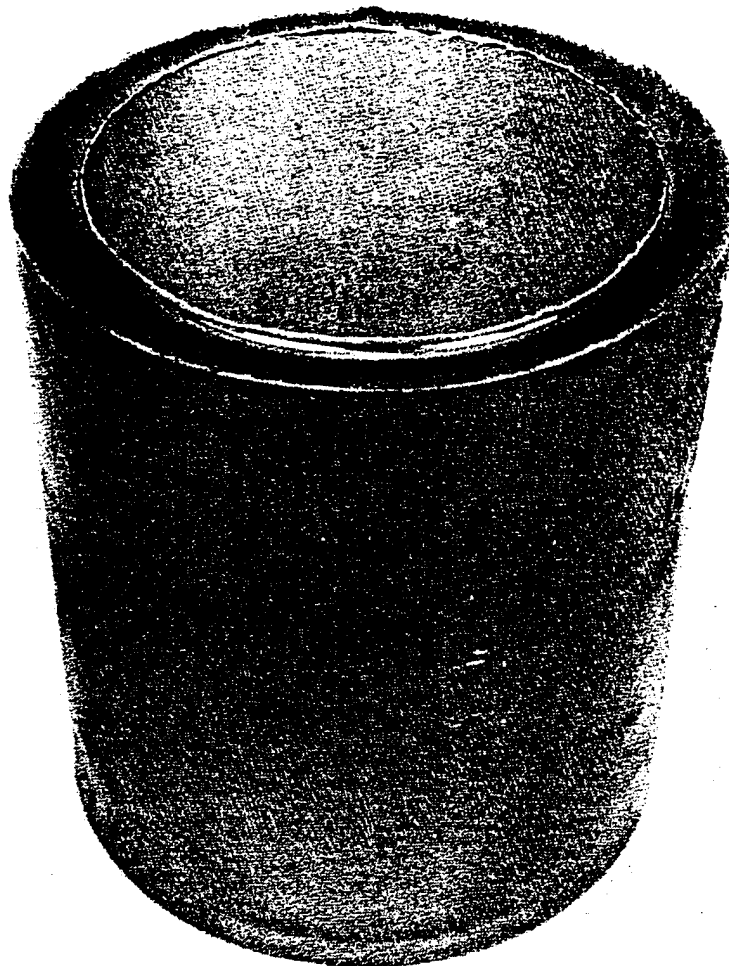
## 1. OVERVIEW

This report describes work on developing a process for casting RBM 2-8-2 polyurethane Pulse Power Decade Model T.C. Output Switch Envelopes. These relatively thick walled hollow cylinders are shown in Figure 1.1. The high energy absorbing and impact and shock resistant elastomeric polyurethane is a candidate for replacing the currently employed polymethylmethacrylate polymer since it is much less likely to shatter from the shock resulting from the energized switch.

RBM-2-8-2 polyurethane is prepared by the reaction of a component system consisting of a prepolymer and chain extender. The process consists of preparing the prepolymer and chain extender, heating, degassing or evacuating and then mixing them, evacuating the mixture, and then pouring the degassed mixture into a preheated mold and applying vacuum again. Details for performing all of the above steps for preparing the casting are described in this report.

The material must be processed in a well ventilated area, but since it does not contain MOCA, also known as MBOCA or as 4,4'-methylene-bis(2-chloroaniline), the processing does not come under the special governmental regulations required specifically for handling MOCA.





0 1 2 3 4 5 6  
LM 0 1 2 3 4 5

Figure 1.1 — Cast RBM 2-8-2 Polyurethane Cylinder for Decade Module T.C.  
Output Switch Envelopes

## **2. MOLD CONSTRUCTION AND AUXILIARY EQUIPMENT**

The mold which is filled with resin up to within about one inch of the top is shown in Figures 2.1 - 2.3. Although Figures 2.2 and 2.3 were previously presented<sup>1</sup> they are also shown here to provide continuity to our description of the mold. The cylindrical walls of the mold were obtained using commercially available sections of aluminum pipe (10.750 in O.D. x 0.365 in thick wall) and a seamless aluminum tube (8.000 in O.D. x 0.250 in thick wall) which are inserted into a grooved aluminum base plate and held in place by screws. Four push-out holes are provided for each of the two cylindrical walls.

In order to allow additional space for the resin to rise while bubbling and foaming during evacuation, we installed easily removable cylindrical sheet metal extensions about 7 in. high onto both the inner and outer cylinders. The sheet metal extension for the outer cylinder is shown in Figure 2.4. A similar extension but with supporting straps along the inside wall is employed for the inner cylinder. Also shown in Figure 2.4 is the resin feed line running to the mold from the resin tank which is supported on an acrylic vacuum chamber lid.

Figure 2.5 shows the vacuum chamber with the resin feed line to the mold. In Figure 2.6 we see a top view of the vacuum tank, the mold with its sheet metal extensions and the feed line which draws resin from the bottom of the bucket.

In one of our castings, as a result of the combination of slight play in the mold and a protrusion of a small quantity of material remaining in one of the baseplate grooves, the cylinder did not align in a parallel manner. This caused that casting to display nonparallel wall. Despite this, material did not leak from the mold,

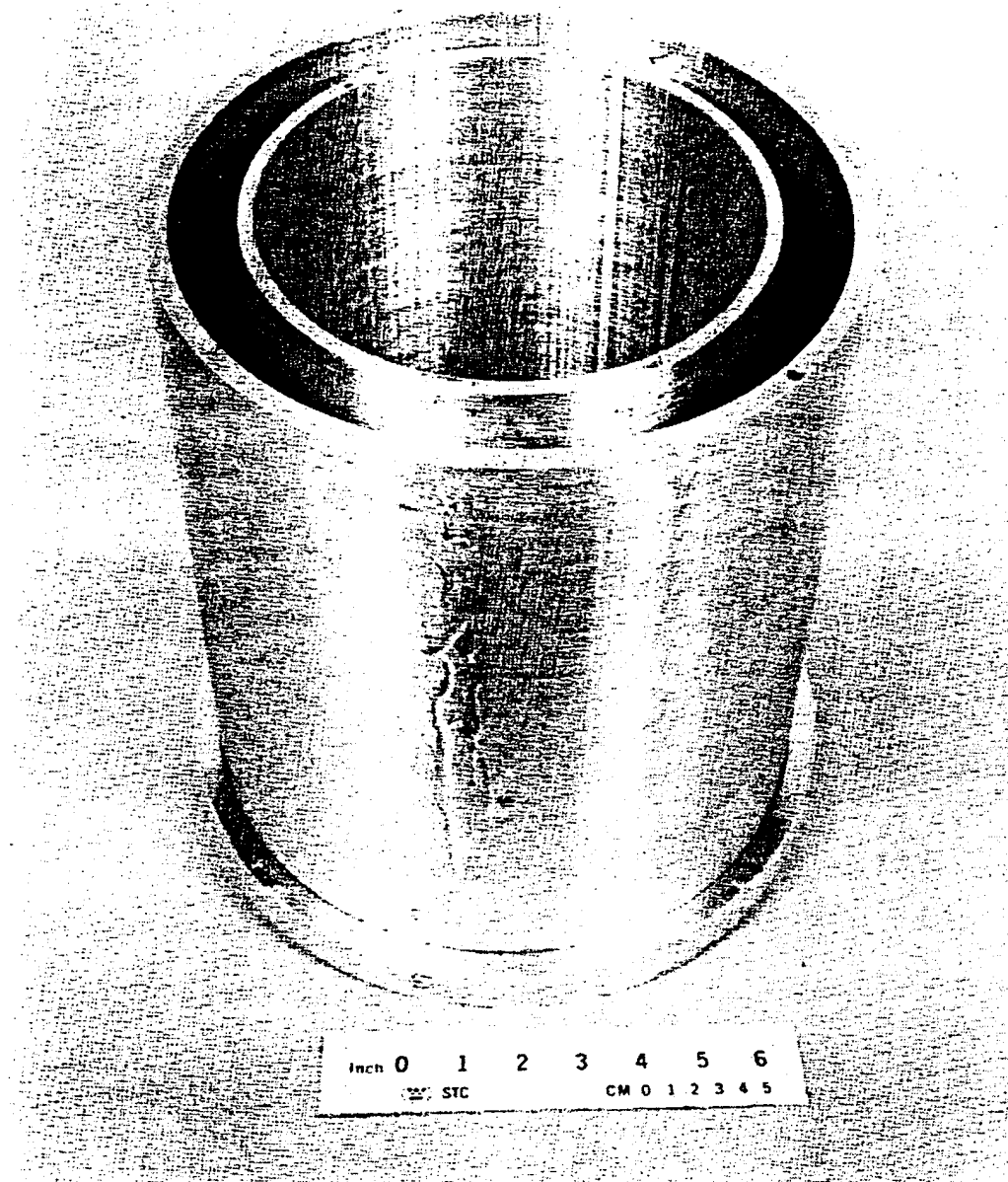


Figure 2.1 — Mold for Cast Polyurethane Cylinder for Decade Module T.C. Output Switch Envelope

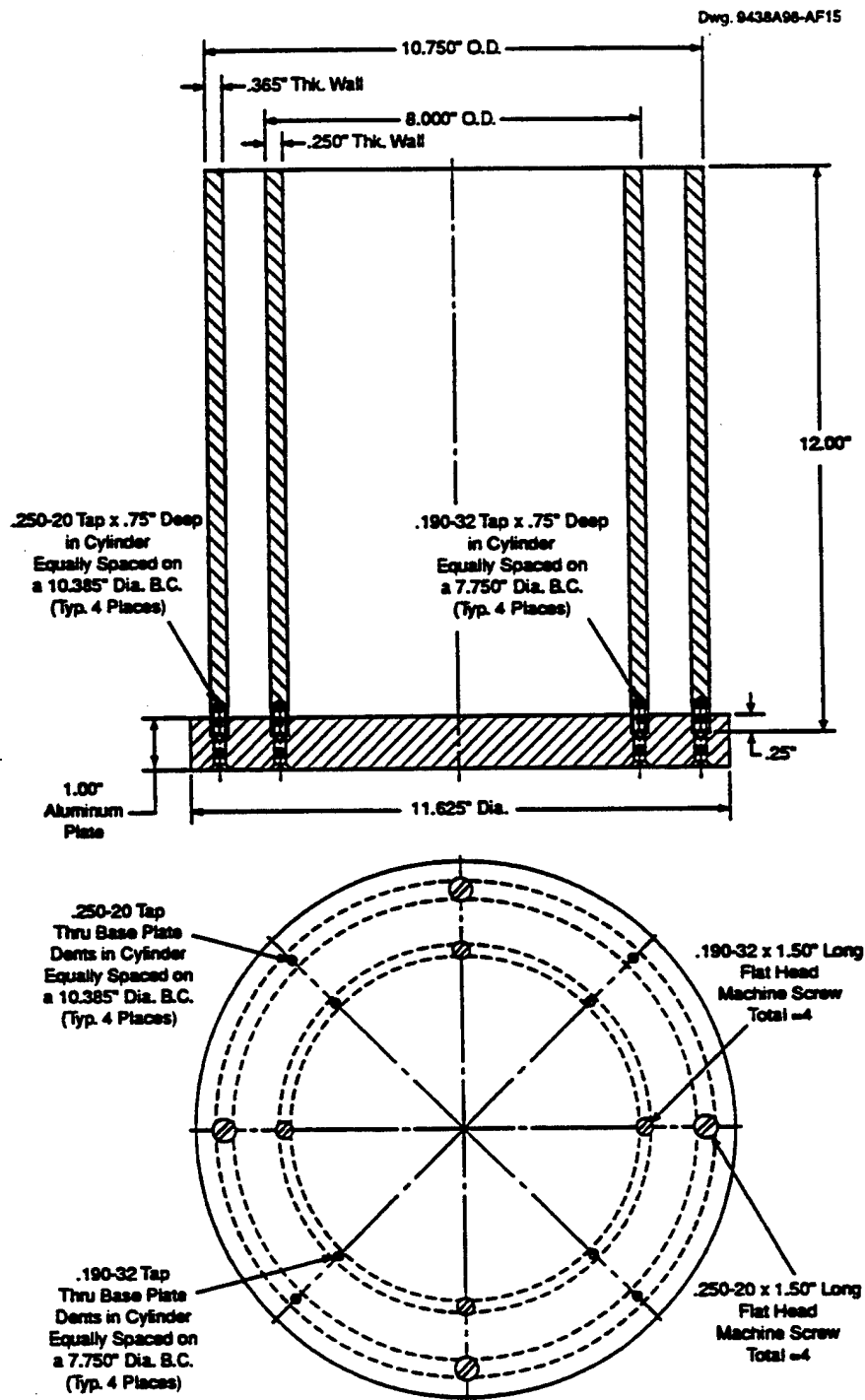


Figure 2.2 — Mold for Cast Polyurethane Decade Module T.C. Output Switch Envelope

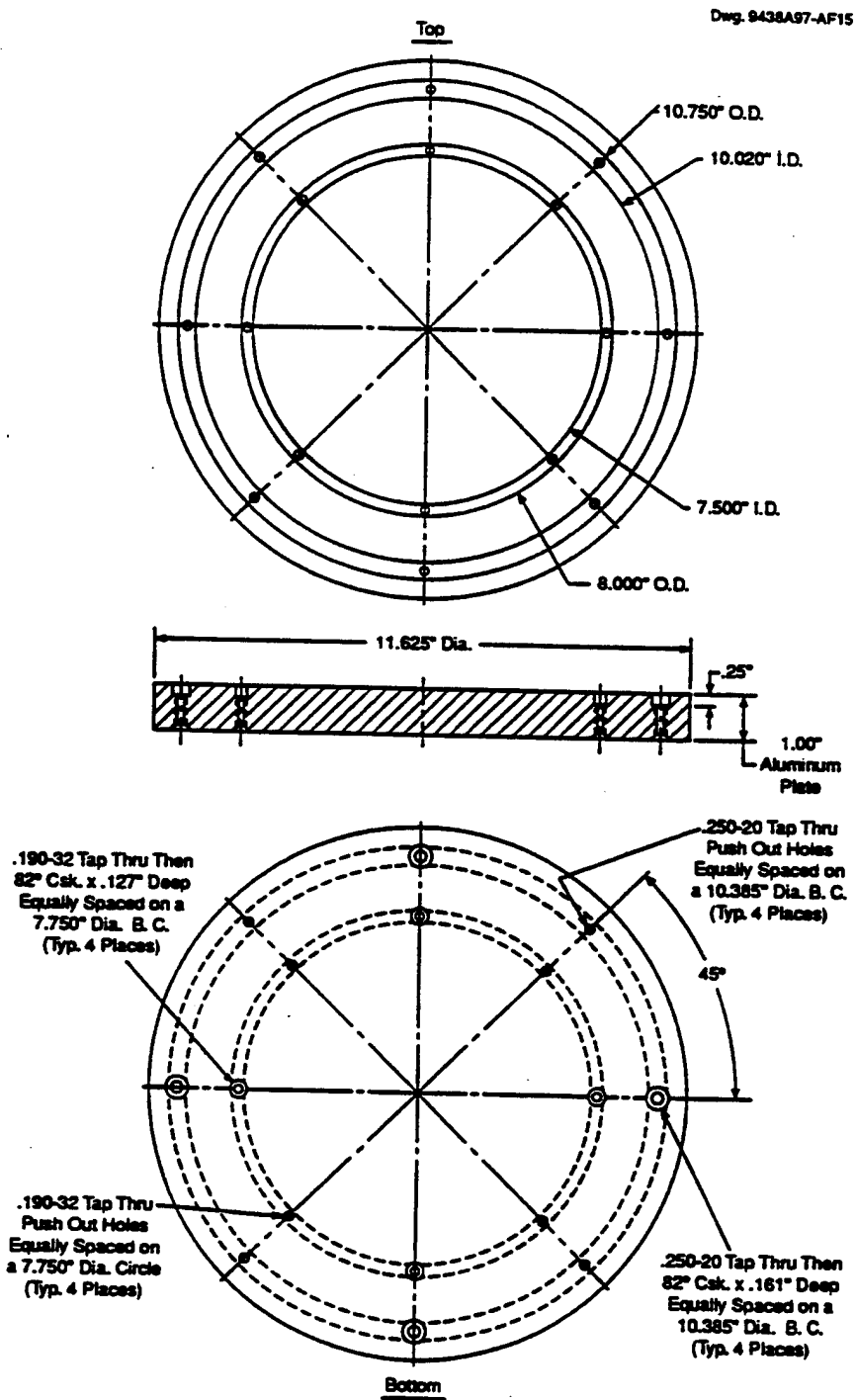


Figure 2.3 — Base Plate for Mold for Cast Polyurethane Module T.C. Output Switch Envelope

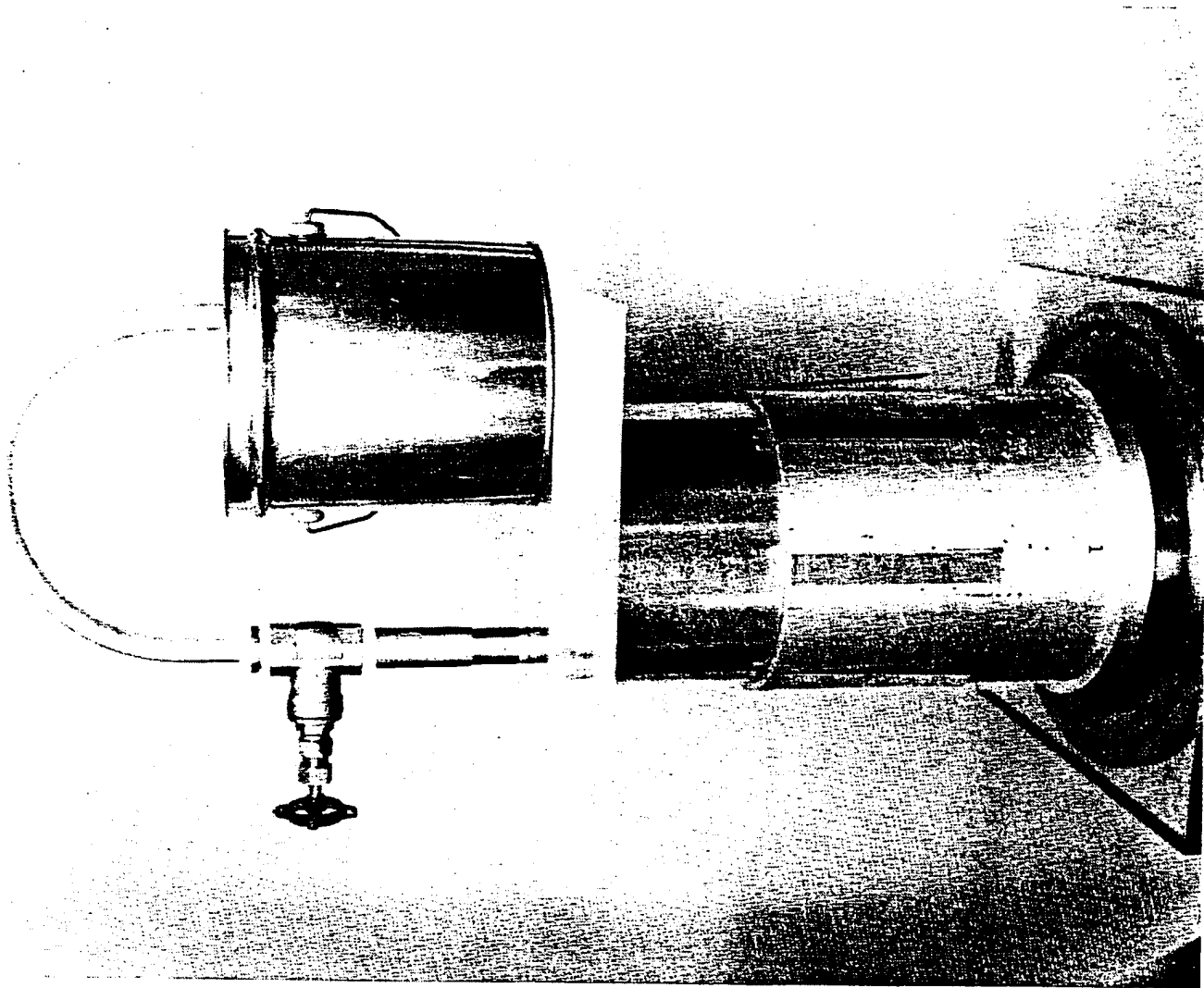


Figure 2.4 - Mold With Sheet Metal Extension, Feed Line From Resin Container to Mold and Acrylic Vacuum Tank Lid

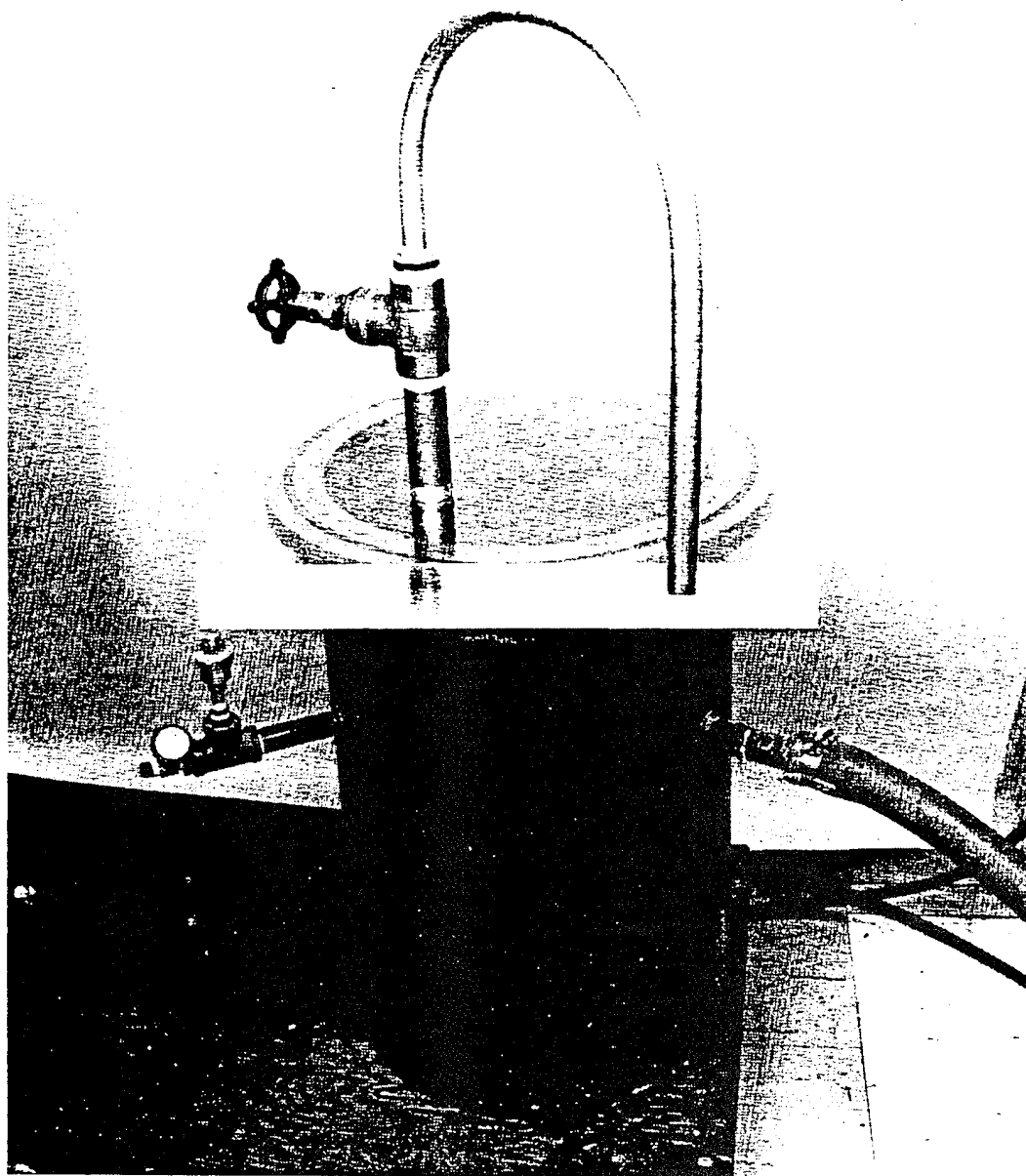


Figure 2.5 — Vacuum Chamber With Lid and Resin Feed Line

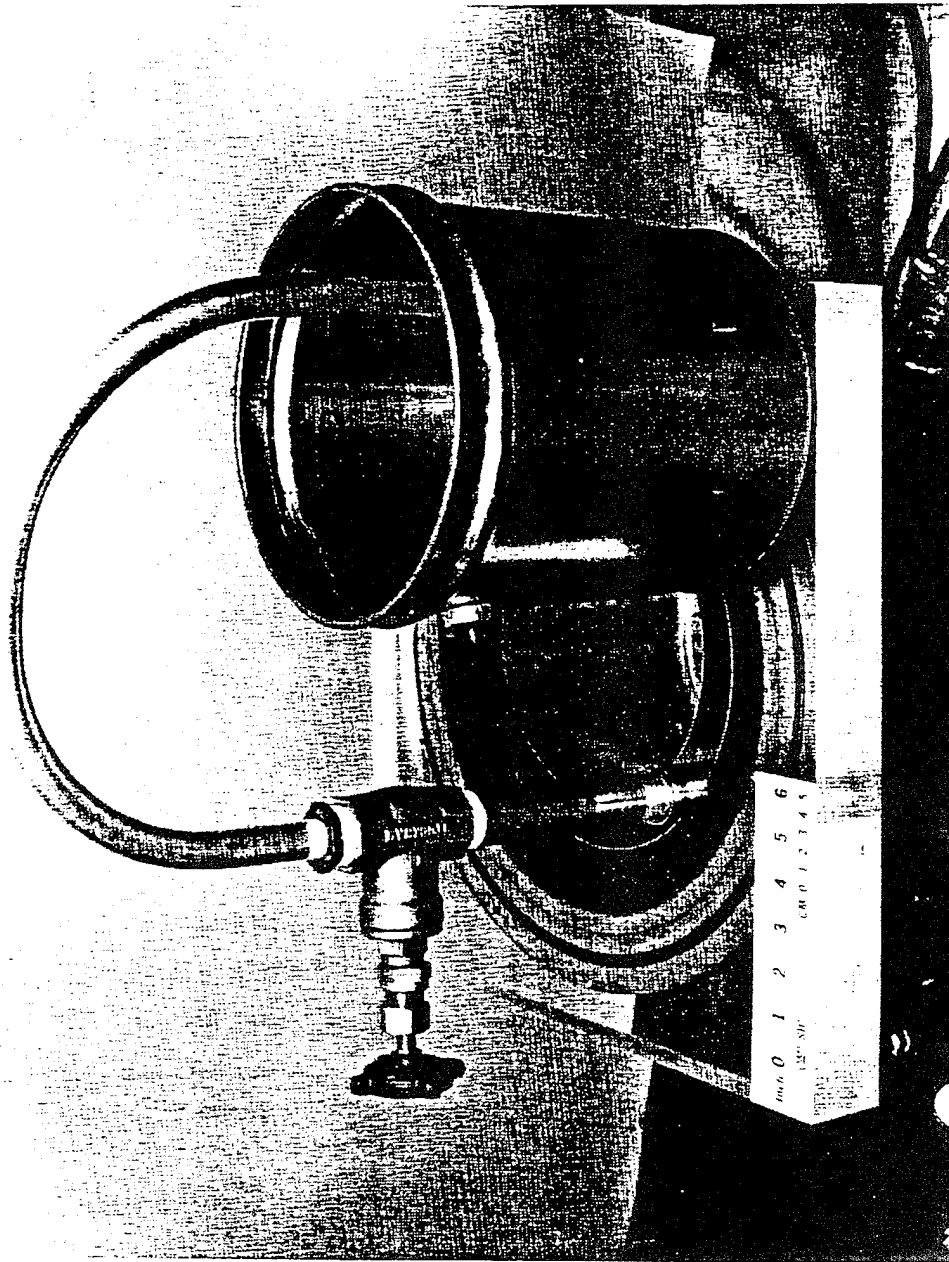


Figure 2.6 - Top View of Vacuum Chamber, Sheet Metal Extensions for Mold, Resin Feed Line and Resin Container



since we apply a silicone RTV sealant where the cylinders enter the grooved baseplate. Besides the obvious necessity of being more meticulous concerning the baseplate grooves and bottoms of the cylinders we introduced spacer bars so as to ensure the parallelism of the two mold cylinders regardless of the mold hygiene. Two spacer bars, 1 in high x 1/8 in thick, at 90° to each other extended diametrically across the top of the mold. Each bar had 1/4 in deep slots with widths corresponding to the cylinder wall thicknesses. The slots were located so as to match the baseplate grooves. At the center, the bottom of one bar and top of the other were notched at least halfway through so as to permit their overlap or crossing. The sheet metal extensions were notched to accommodate the spacers.

Demolding the structure required considerable force because of the roughness of the mold, the high surface area for this relatively large part, and the inability to take much advantage of the different coefficients of thermal expansion between the aluminum and polyurethane since the cylindrically shaped casting was surrounded on both the inside and outside by the metal mold.

In order to demold we first removed the baseplate; however, we found that we could not generate sufficient force using the push-out screws. This was solved by inverting the mold and impacting the base plate over a 4 in x 4 in wooden pedestal. Next we broke loose the outer mold cylinder using a hydraulic press. Once some relative sliding movement occurs it is generally not difficult to push off the outer mold cylinder. The polyurethane is loosened from the inside mold cylinder again by using the hydraulic press, to initiate breaking the bond. Since the casting has shrunk tightly against the inner mold cylinder we find it convenient to continue the separation process using an Arbor press.

For future production purposes we suggest (a) designing the shape and size of the mold so as to reduce subsequent machining costs of the finished part, and (b) employing a smooth mold which would result in easier demolding, a scar free surface, and superior optical clarity. Incorporating a Teflon liner may be helpful.

Increasing the height of the mold so as to allow more rise space during evacuation and providing handles or grips would also be desirable.

Although we have employed rectangular bars for selectively pushing out a given cylinder in the press during demolding, it would be an added convenience and less damaging to the mold to employ circular supports in the form of rings. Appropriately located circumferential recesses in the circular supports could be employed to retain the desired cylinder. Circular plates of the appropriate diameter with positioning recesses could also be quite useful in pushing the mold apart. With a smooth mold and adequately supported aluminum cylinders it may be possible, using a press, to employ a ring to push out the casting simultaneously from both cylinders.

### 3. PROCESS DEVELOPMENT

This Section describes the development of the process for casting the cylindrically shaped envelopes for the Decade Module T.C. Output Switches. The casting is prepared from the reaction product of a polyurethane prepolymer and a chain extender. Although previously provided<sup>1</sup>, detailed descriptions of the chemical compositions and methods of preparation of the prepolymer and extender are presented in Section 4 for the reader with a special interest in this area. The next Section also contains a general description of the chemistry, specifications for the raw materials and suggestions pertaining to quality control.

The first two cylindrically shaped T.C. Output Switch Envelope castings were processed by mixing the prepolymer and extender, evacuating the mixture to remove air bubbles introduced by the mixing and CO<sub>2</sub> generated from the reaction between the isocyanate groups and the small quantity of water present in the mixture as a contaminant. The mixture was then poured into the mold and evacuated again to remove air bubbles introduced during the filling of the mold as well as air entrapped between the base plate and bottom of the mold's two cylinders. Despite these efforts the resultant castings nevertheless contained bubbles.

We then went to a more rigorous evacuation or degassing process, which is described below and basically consists of evacuating the prepolymer and extender just prior to mixing as suggested in Section 4.3, and filling the mold while it is under vacuum. After the prepolymer and extender were brought to their respective premixing temperatures of 100-110°C and 145-150°C they were evacuated at about 5 Torr until the foam broke and then returned to their ovens and heated briefly under dry nitrogen so as to compensate for their slight heat loss during the

degassing step. The next alteration in the process involved entering the mixed and again evacuated material into the bottom of the mold under vacuum using the previously described equipment (Figures 2.4-2.6). The resin feed line was inserted to within about 1/2 in from the bottom of the feed bucket which contained the reacting mixture. The feed line, while externally sealed by means of an O ring against the acrylic lid of the vacuum tank, extended to within about 1/4 in of the bottom of the mold which had been placed inside the tank. With the valve closed the tank was evacuated, and then the valve was slowly partially opened to permit the liquid resin to be drawn into the mold. As the resin entered the mold it bubbled and foamed vigorously at first. Because the height of the mold is only about one inch above the top of the casting we had to enter the material slowly, and intermittently moderate the vacuum so as to prevent the foaming and bubbling resin from overflowing the mold. As the gas was pulled off the foaming and bubbling subsided, and the pressure in the tank was reduced to about 3-5 torr. The filled mold was then removed from the vacuum tank. Inspection of the casting after curing and demolding revealed that it was free of bubbles or voids. In order to reduce the time required to attain a "good" vacuum, we mitigated the bubbling and foaming problem by adding extension pieces to increase the height of the mold. These helped, and again we produced a bubble-free casting.

Now that we developed some confidence that we could produce void-free castings we decided to examine simplifying the process. In essence we added a defoaming agent, filled the mold under atmospheric pressure and performed vacuum degassing when the mold was about one-third full, and again after the mold had been filled. The defoaming agent, BYK-052, manufactured by BYK Chemie, was added as 0.02 weight percent based on the combined weight of the extender and prepolymer. Half of the defoamer was stirred into the prepolymer just prior to its individual evacuation in order to reduce the time required for that step in the process. The remainder of the defoaming agent was added while the extender and prepolymer were being mixed. Degassing the partially filled mold, in order to remove any air entrapped at the bottom of the mold in addition to gas in the resin,

proceeded quite easily requiring only a small fraction of the time previously experienced. This reduced exposure time to vacuum reduces the quantity of unreacted material that is pulled off thereby improving quality control by reducing batch to batch variability in the stoichiometry of the resin. After the mold was filled the system was evacuated again with relative ease. The finished casting appeared bubble-free and indistinguishable from those poured into the mold while it was under vacuum.

An approach that we held in abeyance and does not appear likely that it will be needed for the Decade Module T.C. Output Switch Envelope consists of curing the material under elevated pressure. Such a process step not only reduces the size of the voids but also increases the solubility of the gas, either air or CO<sub>2</sub>, in the resin. On subsequent aging the dissolved gas would slowly leave the system by a diffusion process.

## **4. FORMULATION AND PREPARATION OF RBM 2-8-2 POLYURETHANE**

This Section, an updated version of the detailed procedure<sup>2</sup> for manufacturing RBM 2-8-2, is presented for the reader who is interested in the fine details of the chemical process. A basic generic outline of the chemistry employed in preparing the polyurethane by the two-step reaction sequence is shown in Figure 4.1 for this process.

The prepolymer is prepared by treating a high molecular weight diol with an excess of the diisocyanate. Just before the casting operation the prepolymer is combined with its chain extender. Since the chain extender contains both hydroxyl and amine components, polymerization proceeds through the formation of both urethane and urea linkages. The reacting mixture solidifies in the mold during curing to give the RBM 2-8-2 polyurethane resin.

Firstly, the raw material specifications will be presented. This will be followed by a description of the preparation of the prepolymer and then of its being combined with the chain extender to prepare the final polymer. In addition, discussions are presented herein of quality control and ancillary items such as record keeping, storage of reactive materials, and safety.

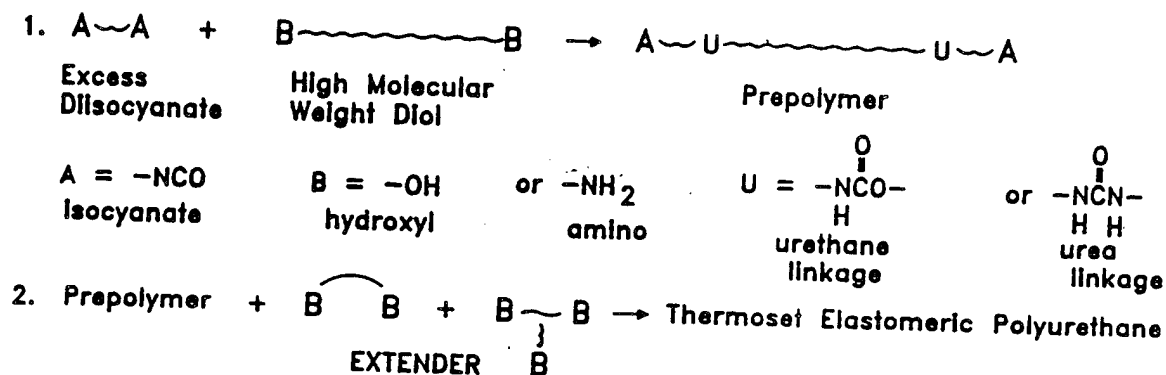
### **4.1 RAW MATERIALS SPECIFICATIONS**

#### **4.1.1 Materials Designations and Procurement Sources**

PTMG 1000, polyoxytetramethylene glycol, ~1000 molecular weight, is available as Polymeg 1000 from QO Chemicals, a subsidiary of Great Lakes

## OVERALL SCHEME

9425A52-AC



## REACTIONS

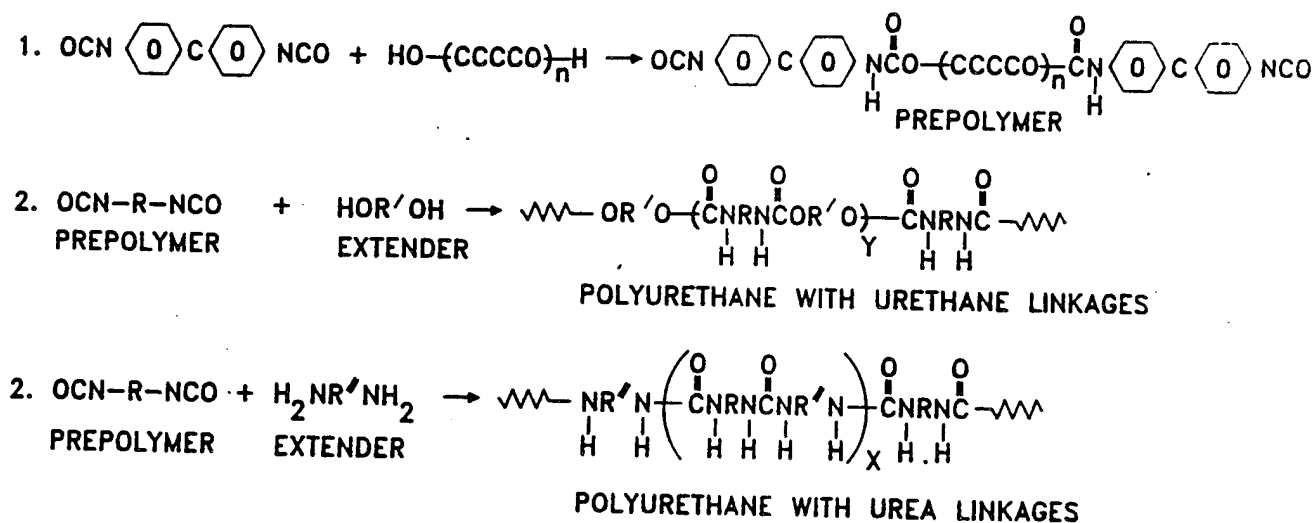


Figure 4.1 — Formation of a two-step polyurethane.

Chemical, or equivalent materials, Terrathane 1000 and Poly THF 1000 are available from DuPont and BASF, respectively.

Desmodur W, methylene-bis(4-cyclohexylisocyanate), is available from Miles.

Dabco T-12, whose principle active component is dibutyltin dilaurate, is available from Air Products and Chemicals.

TMP, trimethylolpropane, is available from Celanese. This compound is also known as 2-ethyl-2-(hydroxymethyl)-1,3-propanediol; 2,2-bis(hydroxymethyl)butanol; 1,1,1-(trihydroxymethyl)propane and 1,1,1-tris(hydroxymethyl)propane.

Versalink 740M Polyurethane Curative (formerly known as Polacure 740M), trimethylene glycol di-p-aminobenzoate, is available from Air Products and Chemicals.

#### **4.1.2 Specified Properties of Chemical Raw Materials**

The procedures for performing the chemical analysis and physical measurements of the raw materials to insure product quality consist of those widely accepted within the realm of analytical chemistry. Each of the suppliers will provide on request references or copies of their particular in-house analytical procedures, product specifications, and Material Safety Data Sheets. This information may be supplemented by the following general references.

D. J. David and H. B. Staley, "Analytical Chemistry of the Polyurethanes",

High Polymer Series, XVI, Part III, Wiley-Interscience, NY, 1969.

ASTM D1638-74, Test for Urethane Foam Isocyanate Raw Materials

ASTM D2849-69, Test for Urethane Foam Polyol Raw Materials

ASTM D2572-70, Determination of Isocyanate Groups in Urethane Materials and Prepolymers



The suppliers' raw material specifications may be verified by employing their in-house test methods, appropriate ASTM test procedures, or the analytical chemistry procedures that are widely accepted and considered in good practice. Substitute raw materials from suppliers other than those listed in this report should, in addition to meeting all of the specified requirements, be in all other respects indistinguishable from the originally described materials. Additional analytical tests for verifying substitutes, as appropriate, should consist of, but not be limited to, the following: infra-red, NMR, chromatography, elemental composition, functional group identification, boiling and melting points and refractive index.

(A) PTMG 1000 Specifications

Hydroxyl Number	110-115
Molecular Weight Dist.	Employ QO or DuPont Specs.
Acid Number	0.05 max
Water, wt%	0.03 max
Volatiles, %	0.1 max
APHA Color	90 max

Acceptance Tests Recommendation

Each lot of QO PTMG 1000 shall meet the above requirements, which should be verified within six months of use. Manufacturer's certified test report is acceptable. The material will be stored under conditions that will not cause any measurable changes in its properties.

The water content and acid number shall be verified within 24 hours of use. If the water content exceeds 0.03 wt. percent, the material may be dried by employing a desiccant, e.g. Drierite or molecular sieves, or by employing heat and vacuum. If a desiccant is employed, the material must be retested and meet requirements for water content, color and acid number. If heat and/or vacuum are

employed, the material must be retested as described above and also meet requirements for the hydroxyl number. Thereafter, the material must be protected from atmospheric moisture, other contaminants and high temperature so that the verified values will not measurably change. The contents of the containers should be sealed under a blanket of dry (dew point  $<-56^{\circ}\text{C}$  ( $-68^{\circ}\text{F}$ )) nitrogen. Throughout this report other inert gases may be substituted for dry nitrogen and all such gases should also have a dew point of  $<-56^{\circ}\text{C}$ .

(B) Desmodur W Specifications

Appearance	Clear water white liquid
NCO Content %	31.8 min
Amine equivalent	132 max
Isomeric Composition	Employ Miles Specs. as of 12/87
Purity %	99.3
Total acidity	0.03 max
Color APHA	50 max
Hydrolyzable chloride	0.04 max

Acceptance Tests Recommendations

Each lot of Desmodur W shall meet the above requirements, which shall be verified within 6 months of use. Manufacturer's certified test report is acceptable. The material will be stored under sufficiently cold and dry conditions (in a sealed container) so that there will be no measurable changes in its properties during storage. If the container had been opened, its contents should be sealed under an atmosphere of dry nitrogen. The isocyanate content shall be verified within 24 hours of use.

**(C) TMP Specifications**

Assay, wt. %	98.0 min
Hydroxyl content, wt. %	37.5 min
Water content, wt. %	0.05 max
Color of 10% Aqueous	5 max
Solution, Pt-Co Units	
Phthalic Color, Gardner	1 max
Acidity as Formic Acid, wt. %	0.002 max

**Acceptance Tests Recommendations**

Each lot of TMP shall meet the above requirements, which shall be verified within six months of use. The manufacturer's certified test report is acceptable. The material will be stored under conditions that will not cause any measurable changes in its properties (according to the manufacturer's specifications).

**(D) Versalink 740M Specifications**

Melting Point	125°C (min. onset of melting)
	128.5°C (max. end of melting)
Water (K.F.)	0.2% max.
Assay (HClO <sub>4</sub> titer)	98% min.
Solubility	Soluble in cellosolve acetate

**Acceptance Tests Recommendations**

Each lot of Versalink 740M shall meet the above requirements, which shall be verified within six months of use. Manufacturer's certified test report is acceptable. The material will be stored under conditions that will not cause any measurable changes in its properties (according to the manufacturer's specifications).

(E) Dabco T-12 Specifications and Typical Properties

Specific Gravity (25°C)	105
Freezing Point	18°C
Boiling Point	Above 200°C at 10 mm
Viscosity, Brookfield (25°C)	41 cs or 43 cps
Flash Point (COC)	235°C
Vapor Pressure	Approx. 150 $\mu$ at 160°C
Total Tin Content (wt. %)*	17.7-18.6
Color (Gardner)*	3 max.
Acid Number* (mg KOH/g sample)	170-177
Refractive Index (25°C)	1.4686
Decomposition Temperature	Above 150°C
Chloride (wt. %)*	0.15 max.
Appearance	Oily liquid (Solid below room temp.)

\*Indicate manufacturer's specifications

Acceptance Tests and Recommendations

Each lot of DABCO T-12 catalyst shall meet the manufacturer's specifications which shall be verified within 6 months of use. The catalyst must display sufficient activity so that during the synthesis of the prepolymer at least 97 percent of the theoretical quantity of isocyanate that should have reacted with the polyol will have been consumed within reaction conditions of 4 hours at 90 to 95°C.

Since the catalyst can hydrolyze on exposure to atmospheric moisture, care must be exercised not to expose it to a humid atmosphere. It should be stored under dry conditions at ambient or lower temperatures.

## **4.2 PREPARATION OF PREPOLYMER**

### **4.2.1 Chemical Composition**

One equivalent PTMG 1000 + three equivalents Desmodur W catalyzed with Dabco T-12, 0.001 percent ;0.0001 percent by weight of the prepolymer.

### **4.2.2 Processing**

#### **(A) Accuracy Requirements**

The quantities of materials, except as otherwise indicated, either in terms of weight or chemical equivalents, used in preparing the prepolymer and casting resin should be within  $\pm 0.10$  percent and temperatures within  $\pm 2.0^{\circ}\text{C}$  of stated values. As employed in this document the term parts refers to parts by weight.

#### **(B) Prepolymer Synthesis**

Just prior to use, the container of Desmodur W should be inspected for crystalline precipitates. If exposed to cool temperatures the trans-trans isomer may precipitate out and should be redissolved by gently warming with agitation. The Desmodur W is then placed in a kettle (batch and kettle sizes are not specified) under a blanket of dry nitrogen ( $< -56^{\circ}\text{C}$  dew point) which is maintained throughout the run.

PTMG 1000 is melted if it is in the solid form. (Its melting point is not sharp and it tends to super cool. The approximate solidification point is  $19^{\circ}\text{C}$ . However, it should not be heated above  $70^{\circ}\text{C}$ . Drums should be rolled during heating so as to minimize exposure time of a portion of the material to elevated temperatures.

At a temperature between  $20$  and  $30^{\circ}\text{C}$ , the PTMG 1000 which contains the Dabco T-12 catalyst is added to the stirred material in the kettle at such a rate that the temperature of the exothermically reacting material does not exceed  $90^{\circ}\text{C}$ . The addition of the PTMG 1000 may be facilitated by cooling the kettle.

After the addition of PTMG 1000 to the Desmodur W has been completed, the contents of the kettle are stirred under dry nitrogen at  $92$  to  $95^{\circ}\text{C}$  for four hours.

The prepolymer, referred to as RBM 2-4, is then degassed for up to 5 minutes at about 5 torr and 90 to 95°C.

On transferring to storage, the prepolymer, which reacts with moisture, must not be permitted to come into contact with ambient air. Containing unreacted Desmodur W, the prepolymer should be stored in clean dry containers having appropriate liners or of stainless steel construction in accordance with the instructions provided in Miles Product Data Bulletin C2-45-1D "Desmodur W", or in a more recently updated bulletin. The void spaces within the storage container should be filled with dry nitrogen prior to sealing.

If stored, the prepolymer should be held under dry conditions at a sufficiently low temperature, less than about 24°C (75°F), so that it will meet the requirements stated below at the time of its use during the casting operation.

(C) Quality Control for the Prepolymer

Samples from each batch of prepolymer RBM 2-4 shall be analyzed and must meet the requirements listed below before the material may be considered acceptable. In addition castings prepared with the prepolymer should meet the physical properties described in the following section on the casting procedure.

<u>Requirements</u>	<u>Test Method</u>
Available Isocyanate Content: 9.2-9.6%	ASTM D 2572
Viscosity at 23°C (Brookfield) 13,000;2500 cps	ASTM D 1638 (except for 23°C temp.)
Physical Properties of Castings on Conversion of Prepolymer, RBM 2-4, to RBM 2-8-2: must meet specs. in the following Section describing the casting process	See Sections 3.2.3 and 4.4

### **4.3 CASTING PROCEDURE FOR RBM 2-8-2 POLYURETHANE**

#### **4.3.1 Pretreatment of the Prepolymer**

The RBM 2-4 prepolymer is placed under dry nitrogen in a forced air oven which is at 140 to 150°C and the prepolymer is brought to 100 to 110°C. During the heating the material is subjected to agitation in order to reduce its heating time and to maintain a uniform temperature thereby preventing localized temperatures in excess of 115°C. As soon as the prepolymer has reached 100 to 110°C (usually within one hour for laboratory size batches, heating of larger size batches can be facilitated by use of continuous operation, an immersion heater or by subdividing the batch) it is degassed at 3 to 4 torr for about 6 minutes for laboratory size batches and about 10 minutes for 11 kg (~25 lb) batches. Alternative procedures can involve processing the RBM 2-4 directly from the kettle in which it had just been prepared or by heating it in an appropriately equipped kettle (contains agitator, heater, gas inlet and outlet) instead of the forced air oven. If a kettle is used the processing conditions should adequately simulate those for the oven.

#### **4.3.2 Pretreatment of Extender**

The premixed extender 4.47 parts TMP and 141.3 parts Versalink 740M, is placed in a forced air oven which is at 155-160°C and agitated under an atmosphere of dry nitrogen. As soon as the extender solution reaches 145 to 150°C, usually in less than one hour, it is degassed at 2 to 4 Torr for about 3 to 4 minutes for laboratory size batches and 4 to 7 minutes for full-scale castings.

#### **4.3.3 Combining the Prepolymer and Extender**

The degassed extender, 0.95 equivalents, at a temperature of 140 to 145°C is poured rapidly into 1.00 equivalents of the agitated prepolymer which is at 100°C. (Weight ratios of extender and prepolymer employed are determined as described in the following subsection). In order to facilitate the rapid pouring of the extender into the prepolymer, essentially the entire preweighed contents of a container of

extender may be emptied; however, this involves compensating for the small residual quantity of material left behind in the container. Should this procedure be used, additional extender equal in quantity to the residue found in prior trial runs may be added to the desired quantity of material prior to its being poured into the prepolymer. An alternative procedure is to weigh while pouring or when near the end of the pour, so as to avoid adjustment for lack of completely emptying the container. Care must be exercised so that the more slowly poured extender does not cool sufficiently so as to cause crystallization and thus phase separation of the amine component.

After the extender has been added to the prepolymer, agitation is continued for about five minutes with care taken so that the resultant mixture is essentially free of bubbles and striations. The material is then degassed under vacuum and poured into molds which are at a temperature of 100°C. If it is desired to remove the polyurethane from the mold, such as for the Physics International insulator rings, the mold should be adequately precoated with a silicone mold release agent. On the other hand where adhesion of the resin to a substrate is desired, great care must be taken to avoid any grease or contaminant on the surfaces to which the resin must adhere. During the casting operations it is recommended that aliquot portions of the mixed prepolymer-extender be poured into test specimen molds for performing appropriate mechanical and electrical tests. ASTM tests described in Section 4.4 had been previously recommended to DNA as a means of quality control.<sup>2</sup>

In the case of molds used for laboratory size quality control test specimens, degassing after filling the mold is optional and usually not necessary if bubbles are not visible. However, if the mold design should result in air pockets or if air enters the liquid reactants during mixing, degassing for several minutes at an absolute pressure of 2 to 5 torr is necessary. Curing is obtained by maintaining the filled mold at 100°C for 20 hours.



#### 4.3.4 Sample Calculation of Material Quantities

##### (a) Stoichiometry

###### Extender

Composition: equivalents  
parts by weight  
Equivalent weight

0.1 TMP + 0.9 Versalink 740M  
4.47 TMP + 141.3 Versalink 740M  
145.77 g/eq

###### Prepolymer

NCO content  
\*Equivalent weight (4200/%NCO)

9.2-9.6%  
446.8 g/eq (assuming 9.40% NCO)

###### Equivalents Ratio (Prepolymer:Extender)

NCO:NH<sub>2</sub>+OH  
\*Ratio of Prepolymer to extender

1.00:0.95  
= 446.8:(0.95 x 145.77)  
= 446.8:138.5

\*Equivalent weight of prepolymer and ratio of prepolymer should be adjusted according to %NCO.

##### (b) Example of Scale-Up to 10,000 g

Scale-Up Factor

$10,000 / (446.8 + 138.5) = 17.085$

###### Quantity of Material

Prepolymer  
Extender

$446.8 \times 17.085 = 7634 \text{ g}$   
 $138.5 \times 17.085 = \underline{2367 \text{ g}}$   
10,000 g

#### 4.4 QUALITY CONTROL TESTS FOR RBM 2-8-2

##### 4.4.1 Pretest Sample Conditions

In order to expedite the evaluation of prepolymer RBM 2-4, mechanical property tests are performed on cast specimens of RBM 2-8-2 on the same day as demolding. In addition, a second group of specimens is permitted to age for 14 to 21 days in an air atmosphere at  $23 \pm 2^\circ\text{C}$  and  $50 \pm 5\%$  RH prior to testing. Mean values should be reported for five replicate specimens for each test.

Specimens that will be subjected to the early tests will be placed in a desiccator over Drierite immediately upon demolding and will be removed individually just prior to testing. This will prevent variable plasticizing effects caused by differences in exposure to moisture in the air. The longer term samples will be permitted to equilibrate with the standard recommended atmosphere and also to continue a slight degree of post-cure.

Although the strength of the RBM 2-8-2 will increase over the 14 to 21 day aging period, both the short-term and long-term specimens should meet the properties shown below.

#### 4.4.2 Test Requirements (previously recommended to DNA<sup>2</sup>)

Compression-3rd Cycle (ASTM D575, specimen ht. - 1.27 cm (0.5 in), dia. - 3.277 cm (1.129 in)).

<u>Deflection, %</u>	<u>Stress, MPa (lb/in<sup>2</sup>)</u>
2	9 ± 3 (1,300 ± 500)
4	18 ± 3 (2,600 ± 500)
6	25 ± 5 (3,600 ± 700)
8	29 ± 5 (4,200 ± 700)
10	32 ± 6 (4,700 ± 800)
inst. set, %	2.5 ± 0.9 (set at instant third cycle load has reached zero)

#### Tensile (ASTM D412, Die C)

<u>Elongation, %</u>	<u>Stress, MPa (lb/in<sup>2</sup>)</u>
10	>15 (>2200)
30	>16 (>2300)
60	>16 (>2300)

100	>17 (>2400)
200	>19 (>2800)
Ultimate	>22 (>3200)
Ultimate Elongation	>230%
Hardness (ASTM D2240)	96-100 Shore A/65-70 Shore D

## 4.5 RECORDS

It is recommended that records of all raw materials, processing conditions and quality control test results be maintained. Performance of the items listed below should be recorded and dated.

### 4.5.1 RBM 2-4 Prepolymer

- a Analysis of raw materials furnished by manufacturer of materials.
- b Verification analysis of raw materials by manufacturer of prepolymer.
- c Thermal exposures of raw materials. This includes temperature and time of storage and warming of ingredients prior to reaction.
- d Quantity of every component entered into kettle.
- e Conditions of reaction. This includes time-temperature data for kettle during entire process, temperature of ingredients during addition to kettle, time for beginning and completing additions of each component, time for presence of dry nitrogen blanket and agitation.
- f Time, pressure and temperature for degassing prepolymer.
- g Analysis of prepolymer.
- h Conditions of storage and shipping of prepolymer (time, temperature, nature of container).

#### **4.5.2 RBM 2-8-2 Casting**

- a Analysis of extender components and quality control tests for prepolymer furnished by suppliers of the materials.
- b Verification analysis by manufacturer of finished casting on percent NCO of prepolymer and, if performed, analysis of the chain extender components.
- c Exposure conditions of prepolymer and chain extender. This includes temperature and time of storage and of warming ingredients prior to reaction.
- d Quantity of prepolymer and extender employed.
- e Conditions of reaction. This includes the temperatures of the prepolymer and extender just prior to mixing, time and temperature data taken at 30 second or shorter intervals during mixing, mold temperature just prior to filling and the mold time-temperature data during cure. In addition the time-temperature-pressure data should be taken during degassing.
- f Mechanical test data of test castings prepared from same master batch of prepolymer-extender mixture. At time of pouring the RBM 2-8-2 into the primary mold, aliquot portions should be poured into test molds for performing the previously described ASTM tests. These molds should be at the same temperature as the production molds, and placed in the same curing oven.

#### **4.6 DELIVERY OF THE PREPOLYMER**

##### **4.6.1 Shipping**

This section applies if the manufacturer of the prepolymer must ship the prepolymer to another plant for the casting operation.

During shipment, the prepolymer should be maintained under cool (less than 25°C (77°F)) and dry conditions. The shipped prepolymer must meet the requirements of Section 4.2 at the time of its use.

#### **4.6.2 Marking**

Each container of prepolymer shall be marked to include the following information:

- (a) Manufacturer's identity and product designation.
- (b) Part drawing number or code designated by procurement agency.
- (c) Lot number and date of manufacture.
- (d) Percent available isocyanate as determined in 4.2.2, part C.

#### **4.7 SAFETY PRECAUTIONS**

The information presented below provides an abbreviated description of some of the hazards and recommended handling and storage procedures. It is not intended as a substitute for studying the manufacturer's literature on storage and handling and Material Safety Data Sheets for each of the materials described in this report.

##### **4.7.1 Desmodur W (methylene-bis(4-cyclohexylisocyanate))**

###### **(a) Personal Contact**

Desmodur W is irritating to the skin upon contact and to the upper respiratory tract. Over-exposure may lead to coughing, headache and shortness of breath, and some individuals may develop allergic sensitivity resulting in asthma-like symptoms. Goggles and/or full face shields and chemical resistant rubber or plastic gloves should be worn. If the concentration of Desmodur W in air is noticeable, canister-type masks equipped for organic vapors or, as necessary, self-contained breathing units should be worn. Safety showers, eye wash stations, polyethylene glycol (300-500 mol. wt.) and adequate ventilation are required during processing. Material Safety Data Sheets such as those issued by Miles should be referred to.

(b) Toxicity Data

From Miles Material Safety Data Sheet on Desmodur W issued 1/24/84.

LD50 Oral Ingestion (Rats)	>11,000 mg/kg (Rats)
LD50 Dermal Single Lethal Dose (Rats)	>10,000 mg/kg
Inhalation 4-Hour LC50 (Rats)	200 mg/m3
TLV	0.02 ppm
Skin Irritation	Irritation upon prolonged contact, skin sensitization and dermatitis possible in some people.
Effects to Eye	Irritating, lachrymatory in high concentrations.
Effects to Lung	Irritant to upper respiratory tract.
Other	Causes allergic sensitivity in some people.
Ames Test	Negative

(c) First Aid

From Miles Material Safety Data Sheet on Desmodur W.

Eye Contact	Flush with water for 15 minutes, consult physician
Skin Contact	Wash affected areas thoroughly with polyethylene glycol (300-500 mol. wt.) and soap and water.
Ingestion	Drink water to reduce corrosivity, consult physician.
Inhalation	Remove to an uncontaminated area, administer oxygen if necessary.

In case of injury a physician should be consulted.

(d) Storage and Handling

Desmodur W must be stored under sufficiently cold and dry conditions so that it will meet the acceptance tests described in 4.1.

Stainless steel or appropriately lined steel containers (c.f. Miles publications on Desmodur W) should be employed.

Spills should be covered with loose, absorbent material (sawdust, dry oil) and tincture of green soap, which contains an alcohol. Material should then be placed in open containers and treated with dilute ammonium hydroxide for 24 hours before disposal. Wash spill area with water and tincture of green soap. Ventilate area to remove vapors.

Fire fighters must wear self-contained breathing apparatus to protect against toxic and irritating vapors. Full protective clothing should also be worn. Avoid water contamination in closed containers or confined spaces because carbon dioxide is involved.

#### **4.7.2 Dabco T-12 Catalyst (dibutyltin dilaurate)**

##### **(a) Personal Contact**

In case of eye contact immediately flush eyes thoroughly with water. All exposed parts of the body and contaminated clothing should be thoroughly washed with soap and water.

##### **(b) Toxicity**

TLV (Skin ACG1H)	0.1 mg Sn/m <sup>2</sup>
LD50 Oral Ingestion (Rats)	3854 mg/kg
Dermal	slightly toxic, highly irritating and will cause burns on skin and eyes

##### **(c) Storage and Handling**

Material can be stored under normal ambient conditions provided it does not come in contact with moisture from the air. If it freezes on storage it can be melted by heating up to 65°C (149°F).

#### **4.7.3 PTMG 10000 (Polyoxytetramethyleneglycol, MW ~1000)**

##### **(a) Personal Contact**

In accordance with good industrial hygiene practice, avoid contact with eyes or skin. Wash after handling. This material has very low toxicity.

##### **(b) Toxicity Data**

From the QO Chemicals Material Safety Data Sheet Polymegs, No. NPVLA 6-70 and Technical Bulletin 207 "QO Polymeg".

LD50 Acute Oral (Rats)	18,830 mg/kg
LD50 Acute Dermal (Rabbits)	>10,250 mg/kg
Eye and Skin Irritation	Minimally Irritating

##### **(c) Storage and Handling**

PTMG 1000 is hygroscopic. Containers should be kept tightly closed to avoid moisture pickup from the air. Drums may be warmed to about 66°C (150°F) for up to 16 hours to permit the PTMG 1000 to melt. Thereafter, storage at 38°C (100°F) will prevent resolidification. However, storage at 100°F should be limited to no longer than 24 hours. When stored in tanks or previously opened containers, the PTMG 1000 must be blanketed with dry nitrogen to prevent moisture pickup, oxidation and possible discoloration.

#### **4.7.4 Polyurethane Prepolymer RBM 2-4**

Since the prepolymer contains unreacted Desmodur W, it should be treated in the same manner as Desmodur W (refer to Section 4.7.1).

#### **4.7.5 Trimethylolpropane (TMP)**

##### **(a) Personal Contact**



In accordance with good industrial hygiene practice, avoid contact with eyes or skin.

(b) Toxicity

TMP is essentially nonhazardous. (Refer to Celanese Chemical Co. Material Safety Data Sheet.)

(c) First Aid

In case of eye contact, flush eyes thoroughly with water.

(d) Storage and Handling

The major consideration in handling trimethylolpropane is that it is hygroscopic and therefore must be protected from moisture. It is manufactured and packaged in a specially dehumidified room to offer maximum protection against pickup of moisture. Trimethylolpropane is packaged in bags and fiber drums. The container is equipped with polyethylene aluminum foil barrier sheets to prevent moisture transmission into the product. Care should be taken in handling the bags to prevent tearing or puncturing, which would affect the protective qualities of the containers. These bags should be stored in protected areas and under conditions of lowest practical humidity, and away from aldehydes, inorganic acids, and isocyanates. Because of its strong tendency to absorb atmospheric moisture, its exposure to normal atmospheric conditions during handling should be minimized.

**4.7.6 Versalink 740M (trimethylene glycol di-p-aminobenzoate)**

Versalink 740M should be employed in a well ventilated area. In accordance with good industrial hygiene, avoid contact with eyes and skin and through inhalation. Wash after handling.

(b) Toxicity

LD <sub>50</sub> (Oral, Vice)	>5000 mg/kg
Dermal	Nonirritating to skin
Eye	Minimal eye irritation

Ames Test                      Nonmutagenic with and without metabolic activation

Effects of Overexposure      Unknown

(c) First Aid

In case of eye contact, flush eyes thoroughly with water and consult a physician. In case of contact with molten material, treat as a thermal injury.

(d) Storage and Handling

Versalink 740M may be shipped in polyethylene lined containers. Storage stability of sealed containers is good under normal ambient conditions. Containers should not be left open to the atmosphere for any period of time to minimize exposure to moisture and oxygen.

## **5. ACKNOWLEDGMENT**

The authors wish to thank Leslie Kennedy for his help in developing the process and preparing the castings.

## 6. REFERENCES

1. M. A. Mendelsohn and L. Mandelcorn, "Impact and Electron Bombardment Resistant Protective Coatings for Pulse Power Diode Insulation - Part 3, Westinghouse Science and Technology Center Report 95-9TC3-PIJUP-R1, February 7, 1995.
2. L. Mandelcorn, et al., "Pulse Power Insulators and Dielectric Development", Westinghouse Science and Technology Center Report 93-9TC3-DNPIN-R1; DNA-TR-92-28, Final Report (Under DNA Contract DNA-001-87-C-0102), February 10, 1993.

**APPENDIX C**

**PROGRESS REPORT ON  
THE CHARACTERIZATION OF  
INSULATOR MATERIALS  
USED IN HIGH ENERGY ACCELERATORS**

To:  
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13 February 1995

By:  
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Dr. M. Kristiansen  
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## **I.Executive Summary**

The goal of this research program is to test the performance of various insulator materials in high vacuum ( $1 \times 10^{-6}$  Torr), high current ( $\sim 300$  kA) surface discharges. Insulator performance is measured in terms of its surface flashover voltage and surface flashover voltage recovery. Other parameters of interest include insulator mass erosion and insulator mass erosion rate. The results are useful in predicting insulator performance in high energy accelerators under fault conditions and insulators used in surface discharge switches and spark gaps.

The insulator materials tested in this report include the "Limitrak" coated polyurethane samples (coated with a new formulation of Limitrak) and the graphitic carbon loaded polyurethane insulator samples. The polyurethane insulator used in all the tests reported in this document is the WC-781 polyurethane. The Limitrak coated samples were coated by Westinghouse with a "chip-resistant" Limitrak formulation of epoxy and 50%  $\text{Al}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$  (i.e., hydrated aluminum oxide) mixture of varying thickness. Due to variations in outside diameter of the coated insulator samples (as much as 0.025"), which was not detected until after the tests, the discharge did not occur on the surface of the insulator and the test data were discarded. The data presented here shows, surprisingly, that under U.V. illumination, the initial surface flashover voltages are higher (up to 30%) than when there is no U.V. exposure. The insulator mass erosion rates are also higher (up to 10%) when the insulator is exposed to U.V. flux. Tests on graphitic carbon loaded polyurethane (1% and 2% graphite by weight), show that the graphitic carbon addition produces no deleterious change in the insulator's surface flashover performance which would indicate that some amount of volume field grading could be accomplished using this type of insulator material.

Modifications of the SDS IV experimental facility vacuum chamber to accommodate the higher surface flashover voltages of the room-temperature cured polyurethane and the experimental tests which expose the insulator surface to e-beam and U.V. radiation fluxes (the e-beam and U.V. flux could promote arcing to the chamber walls) are also shown.

## **II.Experimental Results**

### **Experimental Facility**

The modified SDS IV facility is shown in Figures 1 and 2. The modification consisted of replacing the stainless steel vacuum chamber with a Pyrex (i.e., borosilicate glass) vacuum chamber. Some problems were encountered with the stainless steel vacuum housing when the room-temperature cured polyurethane samples were tested. The surface flashover voltage was high enough that some arcing to the chamber walls occurred. Table 1 lists the operational parameters of the modified SDS IV experimental facility which are unchanged from the original parameters.

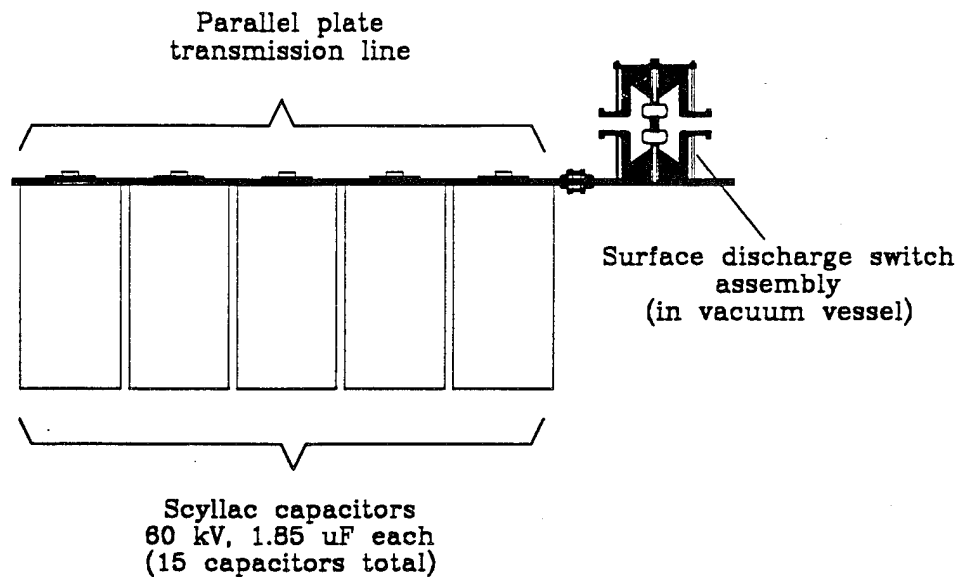


Figure 1. Side view of modified SDS IV experimental facility.

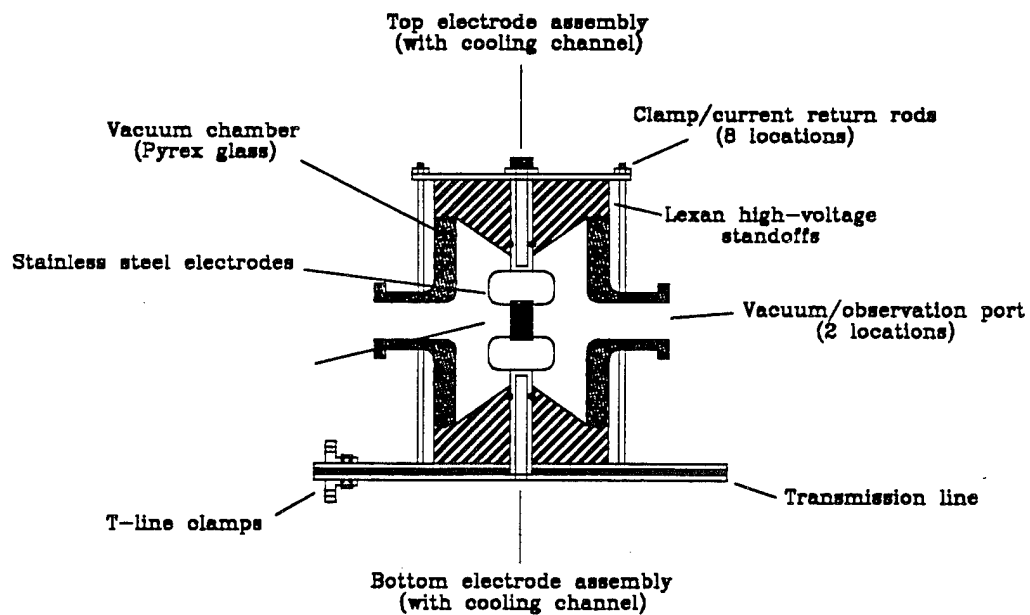


Figure 2. Enlarged view of modified SDS IV experimental facility vacuum chamber.

TABLE 1. OPERATIONAL PARAMETERS OF THE SDS IV EXPERIMENTAL FACILITY.

Parameter	Value
Voltage (maximum)	50 kV
Current (peak, maximum)	600 kA
Oscillation frequency	200 kHz
Effective charge (per discharge)	4.5 C
Energy (maximum)	34.6 kJ
Pressure (minimum)	10 <sup>-4</sup> Torr

### Insulator Test Results

The electrode material used in all the insulator surface flashover tests reported here was stainless steel. The polyurethane insulator material used for the coated and uncoated samples was the WC-781 polyurethane. Figures 3 and 4 illustrate the test results of the Limitrak coated polyurethane with and without U.V. illumination. The U.V. source is a continuously operated, mercury calibration lamp (i.e., 10  $\mu$ W of 253.4 nm radiation at 50 cm). It was noted during these tests that the vacuum chamber was more quickly evacuated to much lower pressures when the U.V. source was in operation. Figures 5 and 6 illustrate the performance of 1% and 2% graphitic carbon loaded polyurethane. The graphitic carbon added to the polyurethane was 325 mesh (i.e., approx. 0.003" diameter particles).

Table 2 lists various experimentally determined parameters of interest for these tests and tests reported in the 26 September 1994 Progress Report. The parameters listed there include the insulator material and test conditions, the breakdown voltages and statistical parameters (highest, lowest, mean, and standard deviation), and the insulator mass erosion rate. The standard deviation listed in Table 2 should not be interpreted in a strict manner since there is insufficient data to support that type of statistical analysis. The standard deviation is used in the context of the present analysis to indicate a level of performance. It is not used for insulator materials that degrade rapidly (e.g., Figs. 3 and 4 illustrate a material that degrades rapidly).

Table 2. Experimentally Determined Polyurethane Performance Parameters.

Insulator Material and Test Conditions	High [kV]	Mean [kV]	Low [kV]	Standard deviation [kV]	Erosion rate [ $\mu$ g / J]
Moca polyurethane, copper electrodes	27.65	16.61	7.00	2.16	0.19
Moca polyurethane, graphite electrodes	27.60	17.28	10.30	2.10	0.17
Moca polyurethane, stainless steel electrodes	24.30	15.20	4.10	2.57	0.15
WC-781 polyurethane, copper electrodes	26.00	15.83	6.90	2.35	0.17
WC-781 polyurethane, graphite electrodes	24.50	16.35	9.10	2.17	0.12
WC-781 polyurethane, stainless steel electrodes	21.50	15.14	7.10	2.97	0.21
Moca polyurethane, "original" limitrak coated, copper electrodes	21.25	16.36	5.50	3.34	0.20
WC-781 polyurethane, "new" limitrak coated, stainless steel electrodes, no U.V.	—	—	—	—	0.42
WC-781 polyurethane, "new" limitrak coated, stainless steel electrodes, U.V.	—	—	—	—	0.44
WC-781 polyurethane, "new" limitrak coated, stainless steel electrodes, no U.V.	—	—	—	—	0.53
WC-781 polyurethane, "new" limitrak coated, stainless steel electrodes, no U.V.	—	—	—	—	0.48
WC-781 polyurethane, 1% graphitic carbon, stainless steel electrodes	27.50	14.03	5.40	1.88	0.16
WC-781 polyurethane, 2% graphitic carbon, stainless steel electrodes	24.30	14.85	7.10	2.10	0.15



## Limitrak Coated Polyurethane (59A series)

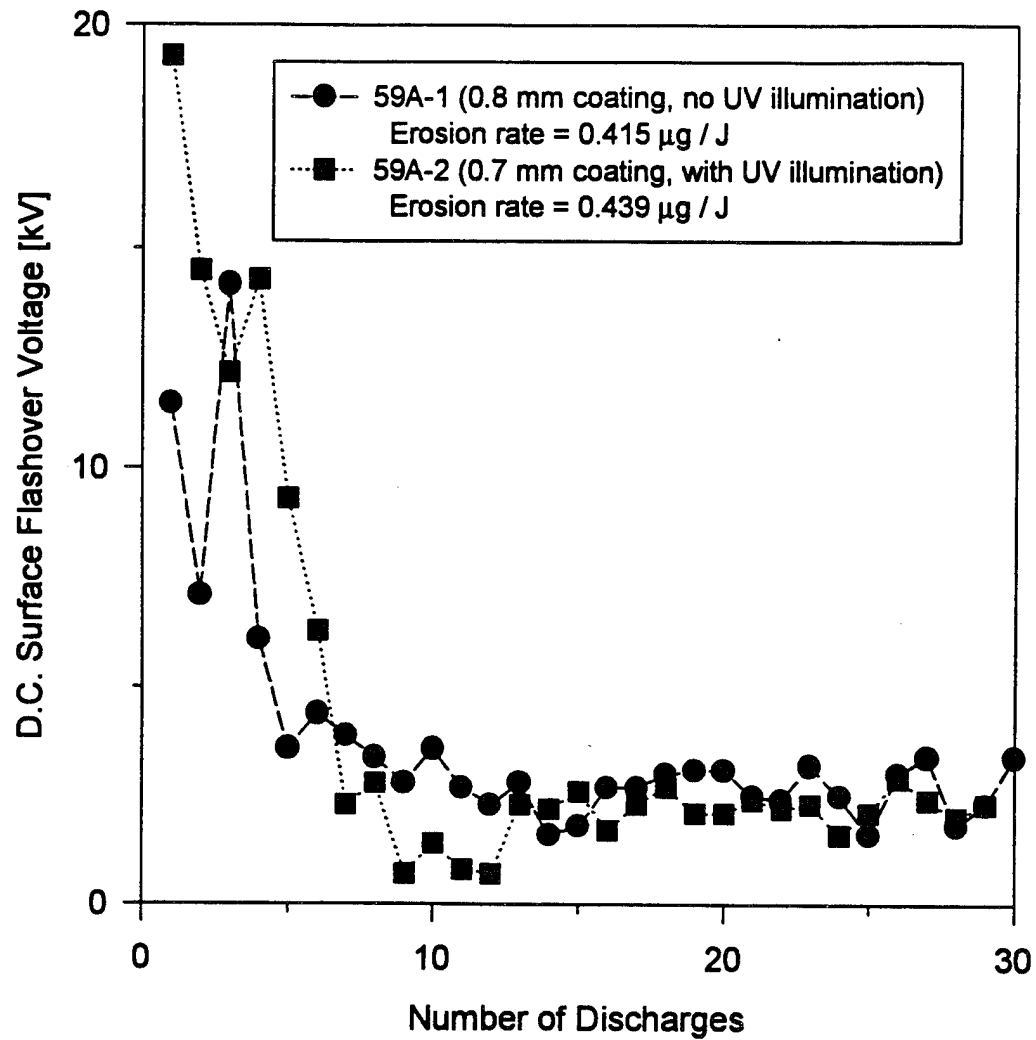


Figure 3. Illustrating the effects of U.V. radiation on the 59A series of Limitrak coated polyurethane.

## Limitrak Coated Polyurethane (67A series)

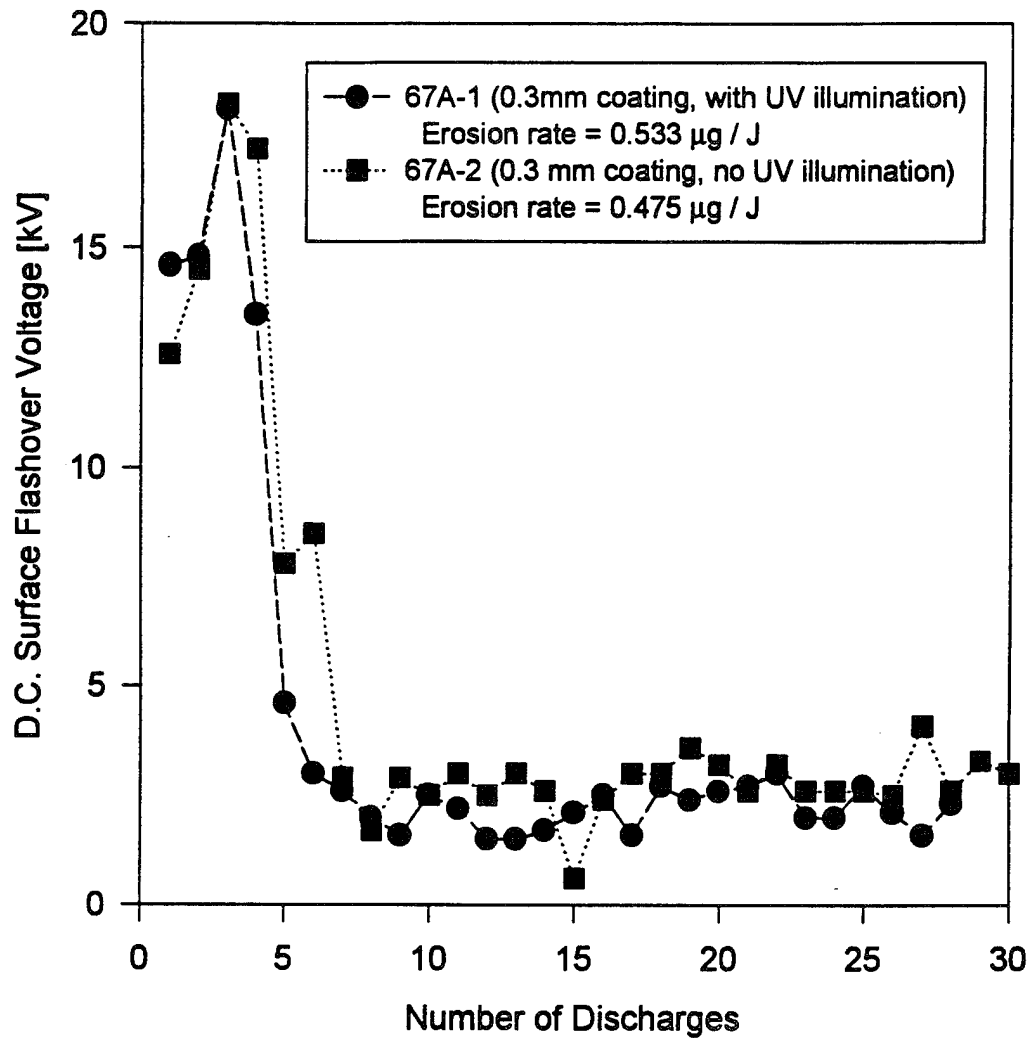


Figure 4. Illustrating the effects of U.V. radiation on the 67A series of Limitrak coated polyurethane.

# **Polyurethane + 1% Graphitic Carbon** (using 325 mesh graphitic carbon)

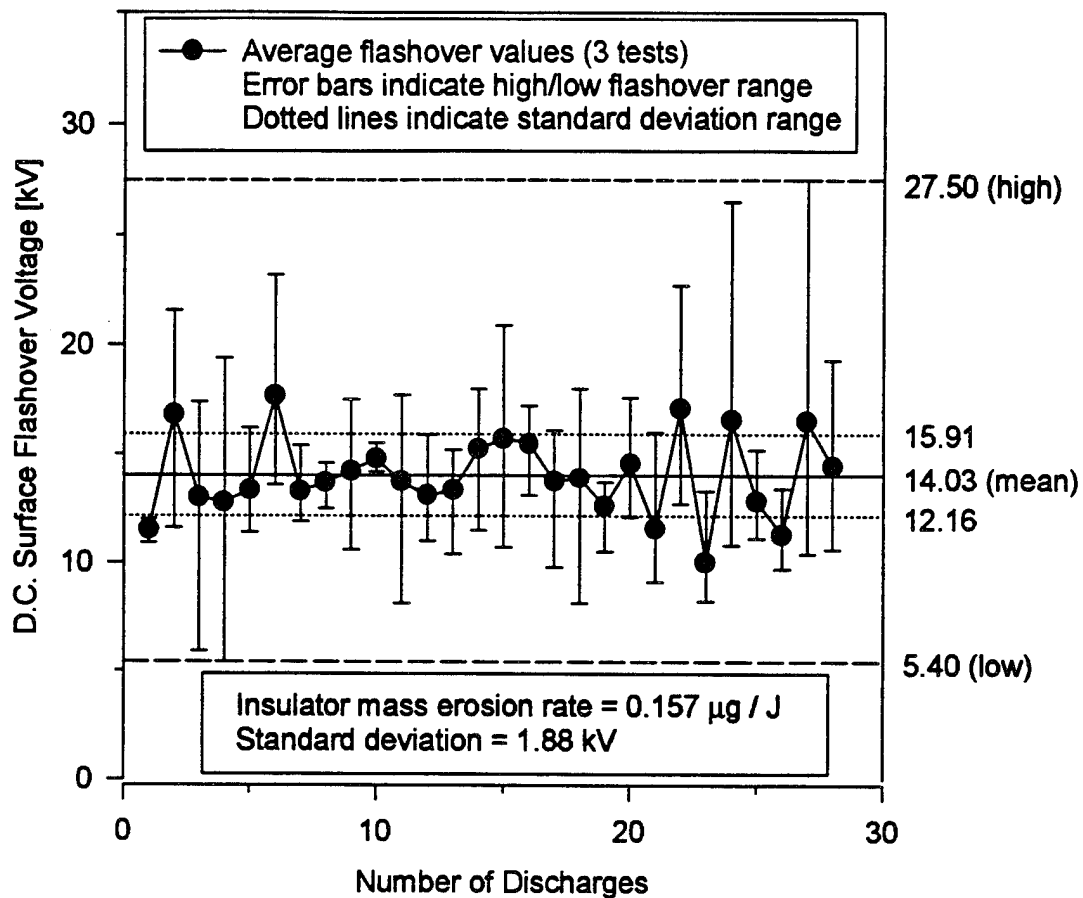


Figure 5. Illustrating the performance of 1% graphitic carbon loaded polyurethane.

**Polyurethane + 2% Graphitic Carbon**  
(using 325 mesh graphitic carbon)

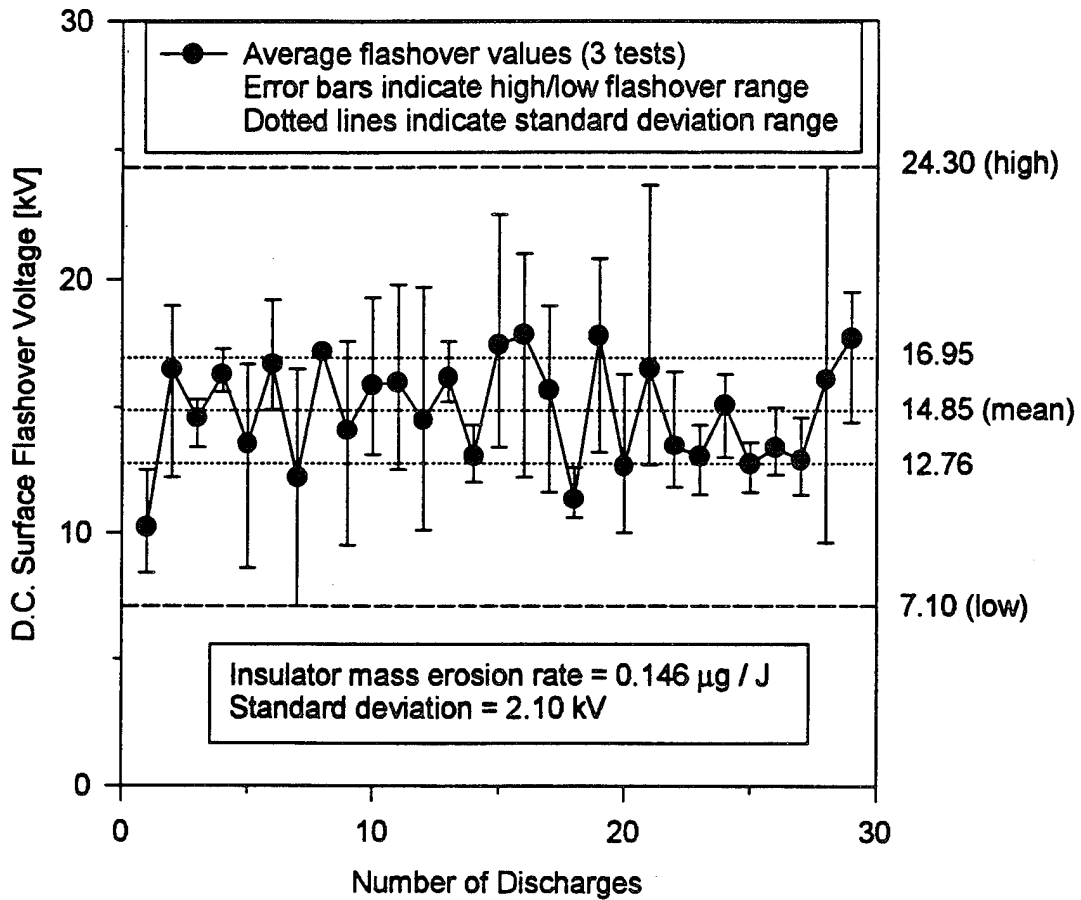


Figure 6. Illustrating the performance of 2% graphitic carbon loaded polyurethane.

### III. Conclusions and Future Investigations

As pointed out in an earlier progress report (dated 26 September 1994), stainless steel electrodes gave the highest standard deviation in surface flashover voltage recovery. This is evident from the data of Table 2 comparing the Moca polyurethane tests versus electrode material. The tests reported here using the WC-781 polyurethane indicate that the standard deviation is significantly reduced by up to 50% when the polyurethane is loaded with 1 and 2% graphitic carbon. The addition of 1% graphitic carbon, however, appears to lower the minimum flashover voltage approximately 25%. The addition of 2% graphitic carbon appears to increase the minimum flashover voltage to its original value. The point to be made is that the addition of graphitic carbon doesn't deleteriously affect the surface flashover properties of polyurethane, which may provide designers a technique to more uniformly grade the volume electric field of insulator stacks.

From Figs. 3 and 4, it can be seen that with U.V. illumination, the initial surface flashover voltage is 30% higher and the mass erosion rate is 10% higher. The data in Fig. 4 is not as dramatic as the data in Fig. 3, but the initial flashover voltage is higher and it does appear to show the same type of trend. Experiments in progress will determine if this presumed U.V. effect is repeatable. At the present time, it is believed that the U.V. illumination removes contaminants (e.g., oil, gas, water) from the insulator surface promoting a higher initial surface flashover voltage. A cleaner surface, however, may possibly lead to higher insulator mass erosion rates since arc energy is not consumed by any surface contaminants and more energy is absorbed by the surface of the insulator. The data of Figs. 3 and 4 also show that the surface voltage holdoff recovery of the new Limitrak formulation is not good and the surface flashover strength of the material degrades rapidly in a few (~ 5) discharges.

The data of Table 2 also indicate that the new, track-resistant Limitrak coating has a substantially higher erosion rate when compared with the original Limitrak formulation and either the Moca-cured or the WC-781 polyurethanes. This could be a disadvantage for larger systems since the increased mass erosion could lead to contamination problems and adverse electrical performance of the insulation.

Tests are now in progress to determine the performance of virgin polyurethane samples and polyurethane samples coated with the chip-resistant Limitrak when exposed to intense electron beams. Following this series of tests will be tests to determine what insulator materials are the better materials for high power switches. If time permits, additional tests will be performed to determine the effects of higher graphitic carbon loading on polyurethane insulators.

### IV. Errata

Minor errors have been found in an earlier progress report dated 26 September 1994. These errors are found in Figs. 5 through 11 of that report. The insulator mass erosion rates were reported in units of  $\mu\text{g} / \text{kJ}$ . The correct mass erosion rates should be  $\mu\text{g} / \text{J}$ . The incorrect erosion rate units also appeared in Table 2 of that earlier progress report.

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